



**Work Plan - Building Interior  
Polychlorinated Biphenyl Supplemental  
Sampling**

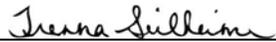
**Madison-Kipp Corporation  
Madison, Wisconsin**

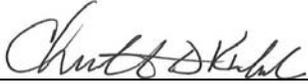
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**Work Plan - Building Interior  
Polychlorinated Biphenyl  
Supplemental Sampling**

Madison-Kipp Corporation  
Madison, Wisconsin

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# Work Plan - Building Interior Polychlorinated Biphenyl Supplemental Sampling

Madison-Kipp Corporation  
Madison, Wisconsin

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**Appendices**

- A Environmental Chemistry Consulting Services PCB and Pesticide Wipe Analysis Supplemental Sample Collection Guidance
- B Pace Analytical Services, Inc. Standard Operating Procedures (S-NY-O-241-rev.05 and S-NY-O-341-rev.02)
- C Anchor Bond Anchor Crete System Specifications

## 1. General

### 1.1 Introduction and Background

On behalf of Madison-Kipp Corporation (MKC), ARCADIS has been retained to conduct additional interior building wipe and indoor air sampling activities for polychlorinated biphenyls (PCBs) at its facility located at 201 Waubesa Street in Madison, Wisconsin (Site). This Work Plan has been prepared and presents the plan for completing wipe and indoor air sampling activities for PCBs in the manufacturing building as discussed during the April 23, 2015, meeting with the Wisconsin Department of Natural Resources (WDNR) and United States Environmental Protection Agency (U.S. EPA). In addition, this Work Plan presents the additional activities coordinated by MKC for employee awareness training and floor cleaning activities.

Below is a chronology of work plans, reports, meetings, and responses from the WDNR and U.S. EPA regarding the investigation and remediation of PCBs.

- On May 31, 2012, a *Site Investigation Work Plan* (Work Plan) was submitted to the (WDNR) for approval to complete site investigation activities at the Site (ARCADIS, 2012a). The WDNR provided a *Conditional Approval* letter dated June 25, 2012, for this Work Plan (WDNR, 2012a).
- On September 28, 2012, a *Site Investigation Work Plan Addendum, Building Subsurface Investigation* (Addendum) was submitted to the WDNR (ARCADIS, 2012b). The Addendum was approved by WDNR in a letter dated October 17, 2012 (WDNR, 2012b).
- On February 14, 2013, a *Building Subsurface Investigation Summary* was submitted to the WDNR to summarize the investigation activities and results (ARCADIS, 2013a).
- On March 15, 2013, a *Site Investigation and Interim Actions Report, February 2012 – January 2013* (SI Report) was submitted to the WDNR to summarize investigation activities and results for the reporting period (ARCADIS, 2013b). On May 29, 2013, a *Supplemental Site Information/Addendum 1* was submitted to the WDNR to provide further information regarding the Site (SI Addendum 1) (ARCADIS, 2013c). The SI Report was reviewed by the WDNR and a response

letter dated June 20, 2013, was prepared that requested a work plan to address "sampling for degree and extent of PCB [polychlorinated biphenyls] and VOC [volatile organic compounds] soil contamination beneath the MKC manufacturing buildings."

- On July 8, 2013, ARCADIS met with the WDNR to discuss the agency's June 20, 2013, response letter and requested a joint meeting with the WDNR and U.S. EPA to clarify the investigation expectations for beneath the manufacturing building.
- On July 23, 2013, ARCADIS met with the WDNR and U.S. EPA to discuss the investigation results completed to date, conduct a site walk, and discuss the objective of additional investigation activities.
- On August 1, 2013, a *Supplemental Work Plan for Polychlorinated Biphenyl Building Subsurface Investigation (Work Plan)* was submitted to the WDNR (ARCADIS, 2013d). The Work Plan was approved by WDNR in the *Madison Kipp Corporation (MKC) Work Plan Reviews* letter dated October 9, 2013 (WDNR, 2013b).
- On April 22, 2014, a *Supplemental Building Interior Polychlorinated Biphenyl Work Plan Subsurface Investigation Summary (SI Report)* was submitted to the WDNR to provide details of the investigation completed from December 2013 through February 2014 (ARCADIS, 2014a).
- On August 27, 2014, ARCADIS met with the WDNR and U.S. EPA to discuss the next steps for addressing the soils containing PCBs beneath the building. At this meeting U.S. EPA requested the completion of indoor air and surface wipe sampling activities, a technical justification submittal for management of PCB contaminated soils beneath the building, and additional soil investigation activities for beneath the building.
- On October 22, 2014, a *Technical Justification – Polychlorinated Biphenyl (PCB)-Impacted Soils Beneath the Main Manufacturing Building (Technical Justification)* was submitted to the WDNR (ARCADIS, 2014b). The Technical Justification included the *Supplemental Work Plan for Polychlorinated Biphenyl Building Subsurface Investigation (Subsurface Work Plan)* as an attachment.

- On November 4, 2014, a *Work Plan for Polychlorinated Biphenyl Building Wipe Sampling* (Wipe Sampling Work Plan) was submitted to the WDNR and U.S. EPA for approval (ARCADIS, 2014c). The WDNR approved the Wipe Sampling Work Plan in electronic correspondence dated December 8, 2014.
- On December 17, 2014, MKC met with the WDNR and U.S. EPA (via telephone) to discuss the Technical Justification and Wipe Sampling Work Plan submittals. During this meeting, U.S. EPA requested continuous soil sampling during the additional soil investigation, PCB homolog analysis for select soil sample locations, and installation and sampling of one monitoring well within the building as part of the Subsurface Work Plan. In addition, U.S. EPA requested preparation and submittal of a Quality Assurance Project Plan (QAPP) for the Wipe Sampling Work Plan. On December 18, 2014, ARCADIS, WDNR, and U.S. EPA participated in a conference call to discuss the proposed QAPP requirements.
- Based on the December 17 and 18, 2014, communications, the Subsurface Work Plan was revised and submitted to the WDNR and U.S. EPA on January 22, 2015, and the *Quality Assurance Project Plan Building Interior Polychlorinated Biphenyl Wipe Sampling* (Wipe Sampling QAPP) was submitted to the WDNR and U.S. EPA on February 19, 2015 (ARCADIS, 2015a). The Subsurface Work Plan was approved by WDNR in electronic correspondence dated January 23, 2015. The Wipe Sampling QAPP was approved by U.S. EPA in electronic correspondence dated February 25, 2015.
- On April 21, 2015, a *Building Interior Polychlorinated Biphenyl Investigation Summary* (Summary Report) was submitted to the WDNR to provide details of the investigations completed in March and April 2015 (ARCADIS, 2015b).
- On April 23, 2015, MKC and ARCADIS met with the WDNR at the MKC facility and U.S. EPA (via telephone) to discuss the Summary Report. During this meeting, U.S. EPA recommended additional wipe and indoor air sampling activities.
- On June 10, 2015, MKC and ARCADIS met with the WDNR and U.S. EPA (via telephone) to discuss additional wipe and indoor air sampling activities recommended during the April 23, 2015, meeting.

This Work Plan has been prepared and divided into the following seven sections:



1. General
2. Objective
3. Schedule
4. PCB Awareness Program
5. Floor Cleaning Activities
6. Sampling Plan
7. Reporting.

## **1.2 Site Location and Description**

The Site is located at 201 Waubesa Street in Madison, Wisconsin. The Site is located in the southwest quarter of Section 5, Township 7 North, Range 10 East in Dane County. The location of the site is illustrated on a topographic quadrangle presented as Figure 1-1.

The Site is approximately 7.5 acres in size. A 130,000-square foot building occupies much of the Site. Asphalt parking lots are located in the northeastern, southwestern and southeastern portions of the Site. The building has a 25,000-square foot second floor and a 25,000-square foot basement. In addition, a 6,000-square foot building is currently being constructed on a portion of the northeast parking lot. Figure 1-2 depicts the layout of the Site. The Site is Zoned M-1 (industrial/manufacturing). The Site is currently used as a metals casting facility.

The Site is located in the eastern portion of Madison, in a mixed use area of commercial, industrial and residential land use. The Site is bounded by a bicycle trail (Capital City Trail) to the north, Atwood Avenue to the south, and Waubesa Street to the west. Residences are located adjacent to the east and west sides of the Site, and further west (across Waubesa Street) and east (across Marquette Street). Commercial properties are located to the south (across Atwood Street) and further east. The Goodman Community Center is located to the north (across the Capital City Trail).



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The Site is also located at the northeast end of the Madison isthmus, approximately 1,500 feet (ft) north of Lake Monona and approximately 6,800 ft east of Lake Mendota. The topography of the Site is relatively flat, with an elevation ranging from approximately 870 to 880 ft above mean sea level. The Site and surrounding area is serviced by municipal water supply and sewerage systems.

### 1.3 Project Contacts

The following project contact information is provided for this QAPP:

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**Work Plan - Building  
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## 2. Objective

This Work Plan presents the means and methods for conducting additional interior building wipe and indoor air sampling activities at the Site as requested by U.S. EPA to evaluate whether potential residuals from historic activities at this facility could pose a risk to current workers through one of the following pathways: inhalation of or dermal contact from potential residuals that may from time to time be exposed by Site activities. Potential residuals will be measured by laboratory analysis of PCBs by Method 8082/8082A (seven Aroclor analyses: Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260) for wipe samples, and Method TO-10A (seven Aroclor analyses: Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260) for indoor air samples. As requested, the interior wipe samples will be collected within established grids as presented herein and two indoor air sampling events will be completed (summer/winter).

Results of the wipe sampling activities will be evaluated per the requirements of the U.S. EPA's *Wipe Sampling and Double Wash/Rinse Cleanup as recommended by the Environmental Protection Agency PCB Spill Cleanup Policy* dated June 23, 1987, revised and clarified April 18, 1991. Environmental Chemistry Consulting Services detection limits for wipe sample laboratory analysis of PCBs by Method 8082/8082A are 0.5 micrograms per wipe (100 square centimeters [cm<sup>2</sup>]) for Aroclors 1016, 1232, 1242, 1248, 1254, 1260, and total PCBs and 1 microgram per wipe (100 cm<sup>2</sup>) for Aroclor 1221. Laboratory analytical results will be compared to the 10 micrograms per 100 cm<sup>2</sup> cleanup level in accordance with the U.S. EPA's *Wipe Sampling and Double Wash/Rinse Cleanup as recommended by the Environmental Protection Agency PCB Spill Cleanup Policy* dated June 23, 1987, revised and clarified April 18, 1991. If analytical results of the wipe samples are above the cleanup level, a plan will be developed to confirm the analytical results, delineate the extent of exceedances, and/or remediate, if necessary. If analytical results are below the cleanup level, then no further actions are required.

Results of the indoor air sampling activities will be evaluated per the National Institute of Occupational Safety and Health (NIOSH) Recommended Exposure Limit (REL) for PCBs. Pace Analytical Service, Inc. (Pace) reporting limits for indoor air sample laboratory analysis of PCBs by EPA Method TO-10A are 0.1 micrograms per polyurethane foam (PUF) for Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260 and total PCBs. Laboratory analytical results will be compared to the 1 microgram per cubic meter cleanup level for total PCBs in accordance with the NIOSH REL. If



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analytical results of the wipe samples are above the NIOSH REL, a plan will be developed to confirm the analytical results, delineate the extent of exceedances, and/or remediate, if necessary. If analytical results are below the NIOSH REL, then no further actions are required.

### **3. Sampling Plan**

The following sections present a description of the work to be completed during the investigation. The contents of this section were prepared in accordance with NR 716.09 Wis. Admin. Code.

#### **3.1 Health and Safety**

Prior to beginning work each day, a “tailgate” health and safety briefing will be held to discuss the activities and identify ways to ensure the health and safety of Site workers. If conditions are encountered during Site investigation activities that differ from those outlined in the health and safety plan, the Site activities will be reevaluated to determine the appropriate actions that will ensure the health and well-being of the workers.

#### **3.2 Wipe Sampling and Analysis Plan**

A standard wipe test is conducted on a non-porous surface (e.g., unpainted metal surface) and uses a 10 by 10 centimeter template which outlines the sampling area<sup>1</sup>. Per the request of U.S. EPA, additional floor samples, some of which may be porous surfaces, will be collected. It is recommended by U.S. EPA that the wipe medium (e.g., gauze pad or glass wool) be of a known size, prepared with 80/20 iso-octane/acetone solution, and sealed in a glass vial until it is used for the wipe test<sup>2</sup>. The Environmental Chemistry Consulting Services *PCB and Pesticide Wipe Analysis Supplemental Sample Collection Guidance* is attached for reference as Appendix A.

Per the request of U.S. EPA, the additional floor wipe sampling activities will use a 33- by 33-ft grid pattern which will be within the original 100- by 100-ft grid pattern used during the March 2015 sampling activities. Floor samples will be collected from each 33- by 33-ft grid with the exception of where floor samples have already been collected during previous sampling activities (i.e.; Grids 1e, 2a, 2e, 3a, 4d, 5f, 6a, 7c, 8d, 9c, and

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<sup>1</sup> How to Test for PCBs and Characterize Suspect Materials;  
<http://www.epa.gov/pcbsincaulk/guide/guide-sect3.htm>

<sup>2</sup> Title 40: Protection of Environment, PART 761-Polychlorinated Biphenyls Manufacturing, Processing, Distribution in Commerce, and Use Prohibitions, Subpart G-PCB Spill Cleanup Policy, 40 CFR 761.123;  
[http://www.ecfr.gov/cgi-bin/text-idx?SID=2df8d68ef63d748da0318d2bd636ee18&node=se40.31.761\\_1123&rgn=div8](http://www.ecfr.gov/cgi-bin/text-idx?SID=2df8d68ef63d748da0318d2bd636ee18&node=se40.31.761_1123&rgn=div8)

10e). In addition, floor sample collection will be limited to the manufacturing portion of the building and will not be conducted within the delineated floor area to be remediated as shown on Figure 3-1.

Sixty-five wipe samples will be collected from the floor surface in the building. The proposed approximate locations of these wipe samples are depicted on Figure 3-1; however, these are subject to change based on visual observations. Wipe samples will be collected by MKC and submitted to Environmental Chemistry Consulting Services (a state of Wisconsin certified laboratory) for laboratory analysis of PCBs by Method 8082/8082A (seven Aroclor analyses: Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260). Sampling will be completed as follows:

- Collect one floor wipe sample from each 33- by 33-ft grid, within the original grid layout of Grids 2, 4, and 6-10, with the exception of where floor samples have already been collected during previous sampling activities, for a total of 55 floor wipe samples.
- Collect one floor wipe sample from each 33- by 33-ft grid, within the original grid layout of Grids 1, 3, and 5, with the exception of the delineated floor area to be remediated as shown on Figure 3-1, for a total of seven floor wipe samples.
- Collect three duplicate wipe samples which will consist of a “double-wipe” of the investigative location. A separate wipe medium will be used for the duplicate sample. The duplicate sample will be collected in the same manner as the original wipe sample and will be clearly labeled as the “double-wipe” for the investigative location<sup>3</sup>. The location of the duplicate samples will not be labeled on the chain of custody for the laboratory, but will be identified in the letter report.
- The sample area will be wiped in a serpentine pattern both horizontally and vertically<sup>4</sup>. The goal for this type of pattern is to thoroughly wipe the entire sample area twice in a different direction and orientation<sup>3</sup>.

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<sup>3</sup> June 1987 “Wipe Sampling and Double Wash/Rinse Cleanup as recommended by the Environmental Protection Agency PCB Spill Cleanup Policy”;

<http://www.epa.gov/epawaste/hazard/tsd/pcbs/pubs/wipe-samp.pdf>

<sup>4</sup> EPA/600/R-12/051 September 2012 “Polychlorinated Biphenyls (PCBs) in School Buildings: Sources, Environmental Levels, and Exposures”;

[http://www.epa.gov/pcbsincaulk/pdf/pcb\\_EPA600R12051\\_final.pdf](http://www.epa.gov/pcbsincaulk/pdf/pcb_EPA600R12051_final.pdf)

- All wipe samples will be analyzed immediately upon submittal to the laboratory and produce results in units of micrograms per centimeters squared.
- The sampling supplies provided by the laboratory include a prepared sterile gauze pad with 80/20 iso-octane/acetone solution and sample jar. The sampler uses the prepared gauze pad and wipes the sample area as described above. The gauze pad is then folded and placed in the sample jar for transport to the laboratory. Although standard methods are not available for wipe sampling, Standard Operating Practices will be based on the relevant U.S. EPA and Occupational Safety & Health Administration documents<sup>5</sup> and the laboratory-provided sample collection guidance in Appendix A.

### **3.3 Indoor Air Sampling and Analysis Plan**

Per the request of U.S. EPA, additional indoor air sampling activities will be conducted within the manufacturing building. Three indoor air samples will be collected during each of the two additional sampling events. The first sampling event will be conducted when outside temperatures are greater than 85 degrees Fahrenheit. The second sampling event will be conducted when outside temperatures are less than 10 degrees Fahrenheit. The samples will be collected from the same three locations identified during the April 2015 sampling activities and are shown on Figure 3-1.

Indoor air samples will be collected by ARCADIS field staff and MKC and packaged, put on ice, and submitted to Pace for laboratory analysis of PCBs by EPA Method TO-10A (seven Aroclor analyses: Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260). Sampling will be completed as follows:

- Collect indoor air samples via low-volume air sampler and PUF sorbent cartridge.
- Collect indoor air samples over 8 hours during the first shift of the workday, estimated to be from 0800 to 1600 hours.

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<sup>5</sup> EPA/600/R-07/004 January 2007 "A Literature Review of Wipe Sampling Methods for Chemical Warfare Agents and Toxic Industrial Chemicals and OSHA Evaluation Guidelines for Surface Sampling Methods";  
<http://www.osha.gov/dts/sltc/methods/surfacesampling/surfacesampling.html>

- Standard Operating Practices will be based on the U.S. EPA Compendium Method TO-10A Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume PUF Sampling Followed by Gas Chromatographic/Multi-Detector Detection<sup>6</sup> and the laboratory-provided standard operating procedure in Appendix B.

### **3.4 Management of Investigative-Derived Wastes**

Investigative-derived wastes are not expected to be generated during the investigation activities.

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<sup>6</sup> EPA/625/R-96/010b January 1999 "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-10A Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD)"; <http://www.epa.gov/ttnamti1/files/ambient/airtox/to-10ar.pdf>

#### **4. PCB Awareness Program**

A PCB awareness program is currently being implemented for MKC employees and contractors by MKC. The training session includes an overview of PCBs, the regulatory considerations surrounding their use, and the nature and extent of PCB concentrations detected at the Site. The training session also covers the personal protective equipment and waste management standard operating procedures for MKC. Training sessions will be held for new employees and annual refresher training will be provided. Training was completed for personnel at the main plant on May 7, 2015, for personnel at the Sun Prairie plant on May 14, 2015, and for personnel at the north plant on May 28, 2015. Training for contractors is being conducted as needed.

PCB markers were installed to inform personnel that there are PCBs present that require special handling and disposal in accordance with 40 CFR 761. MKC personnel will inspect the PCB markers on a monthly basis to verify markers are intact, in good condition, and that information is visible. The location of the PCB markers are shown on Figure 5-1.



## **5. Floor Cleaning Activities**

Routine interior floor cleaning activities are currently being implemented by MKC. The floor cleaning will be conducted by 40 hour HAZWOPER trained personnel with dedicated floor scrubbers and accessories. All waste streams generated from the floor cleaning activities will be disposed of with a licensed disposal facility.

Per the June 10, 2015, telephone conference, U.S. EPA approved MKC implementing a urethane fortified cementitious coating of select floor areas within Grids 1 through 5, as shown on Figure 5-1. This method has been selected for implementation at this site in lieu of the double wash/double rinse/double epoxy due to the manufacturing activities that are ongoing within the facility. The select floor areas will be power-washed and scarified as necessary prior to applying the coating. One coat will be applied via hand-trowels and will be ¼-inch in thickness. MKC personnel will inspect the floor coating on a monthly basis to verify coating is intact and in good condition. The floor coating activities are tentatively scheduled for mid-August. The Anchor Bond Anchor Crete System specifications are attached for reference as Appendix C.

## **6. Schedule**

Following approval of this Work Plan by U.S. EPA and WDNR, it is planned that the activities will be initiated in stages over the coming months.

- The PCB awareness program and floor cleaning activities are currently being implemented at the MKC facility.
- The floor coating (urethane fortified cementitious coating) of select floor areas is currently scheduled for mid-August.
- The wipe sampling activities will be performed over the course of approximately two days, following which samples will be submitted for laboratory analysis. Laboratory analytical results are expected two weeks following sample submittal.
- The indoor air sampling activities will be performed over the course of one day for each event in the summer and winter months, following which samples will be submitted for laboratory analysis. Laboratory analytical results are expected two weeks following sample submittal.

Analytical results and floor coating documentation will be provided to the WDNR and U.S. EPA as presented in Section 7.0 Reporting.



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## **7. Reporting**

Following receipt of the wipe and indoor air sample analytical results (summer event) and implementation of the floor coating, ARCADIS will prepare a letter report. The letter report will include a summary of the activities completed and the analytical results, and provide recommendations if needed. Copies of the laboratory analytical reports will be included as attachments to the summary letter. The indoor air sample analytical results for the winter event will be provided upon receipt.

## **8. References**

ARCADIS. 2012a. Site Investigation Work Plan. May 2012.

ARCADIS. 2012b. Site Investigation Work Plan Addendum, Building Subsurface Investigation. September 2012.

ARCADIS. 2013a. Building Subsurface Investigation Summary. February 2013.

ARCADIS. 2013b. Site Investigation and Interim Actions Report February 2012-January 2013. March 2013.

ARCADIS. 2013c. Supplemental Site Information/Addendum 1. May 2013.

ARCADIS. 2013d. Supplemental Work Plan for Polychlorinated Biphenyl Building Subsurface Investigation. August 2013.

ARCADIS. 2014a. Supplemental Building Interior Polychlorinated Biphenyl Work Plan Subsurface Investigation Summary. April 2014.

ARCADIS. 2014b. Technical Justification – Polychlorinated Biphenyl (PCB)-Impacted Soils Beneath the Main Manufacturing Building. October 2014.

ARCADIS. 2014c. Work Plan for Polychlorinated Biphenyl Building Wipe Sampling. November 2014.

ARCADIS. 2015a. Quality Assurance Project Plan Building Interior Polychlorinated Biphenyl Wipe Sampling. February 2015.

ARCADIS. 2015b. Building Interior Polychlorinated Biphenyl Investigation Summary. April 2015.

WDNR. 2012a. Conditional Approval: May 2012 Site Investigation Work Plan. June 2012.

WDNR. 2012b. September 28, 2012 Site Investigation Work Plan Addendum: Building Subsurface Investigation. October 2012.



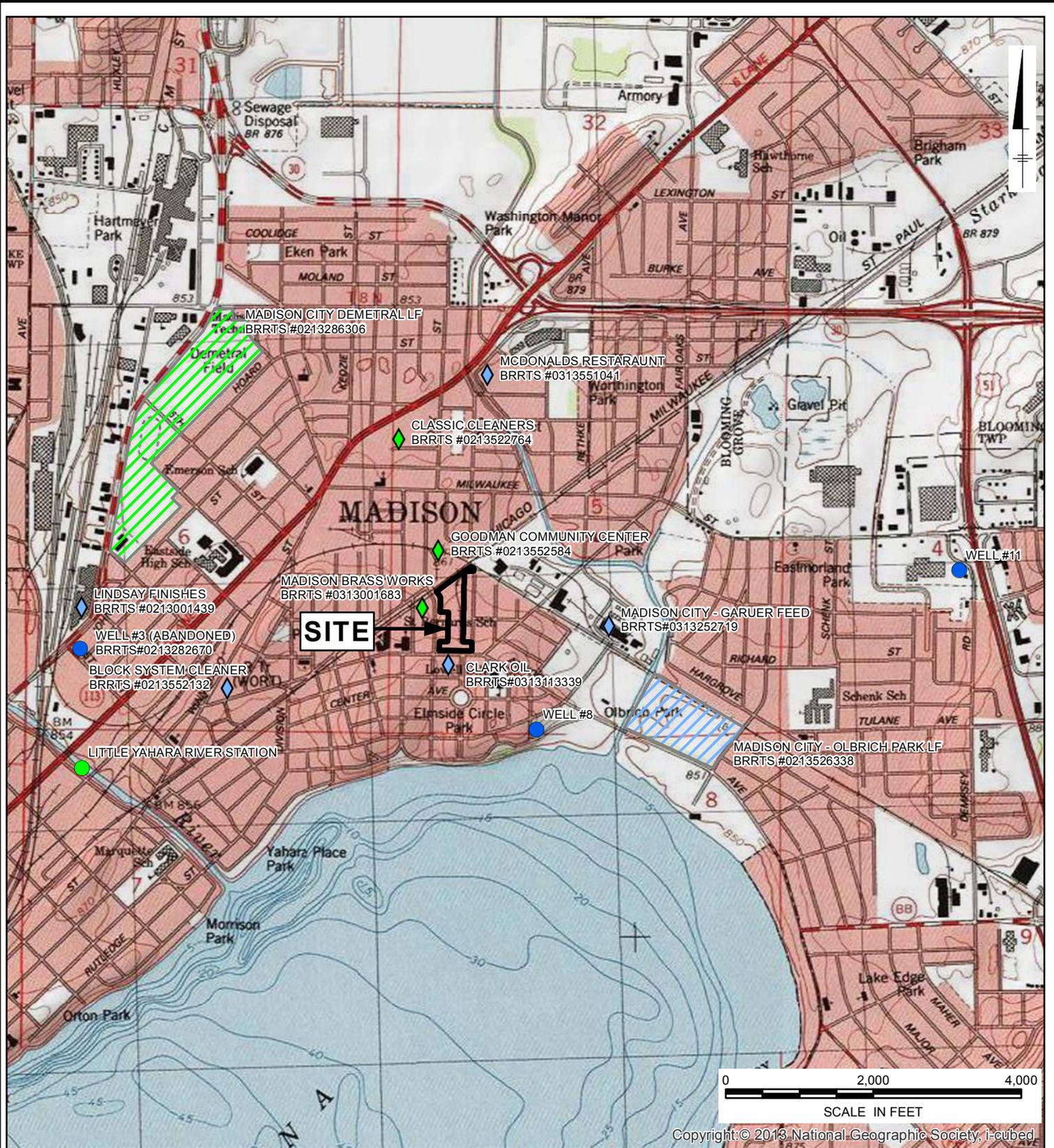
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Madison-Kipp Corporation  
Madison, Wisconsin

WDNR. 2013a. Review of March 2013 Madison Kipp Site Investigation and Interim Actions Report February 2012 – January 2013.

WDNR. 2013b. Madison Kipp Corporation (MKC) Work Plan Reviews. October 2013.

## Figures



**LEGEND**

- ◆ OPEN SITE (ONGOING CLEANUP)
- ▣ OPEN SITE - SITE BOUNDARIES
- ◆ CLOSED SITE (COMPLETED CLEANUP)
- ▣ CLOSED SITE - SITE BOUNDARIES
- GAUGING STATION
- MUNICIPAL WATER SUPPLY WELL
- ▣ PROJECT BOUNDARY



MADISON-KIPP CORPORATION  
201 WAUBESA STREET  
MADISON, WISCONSIN

**WORK PLAN**

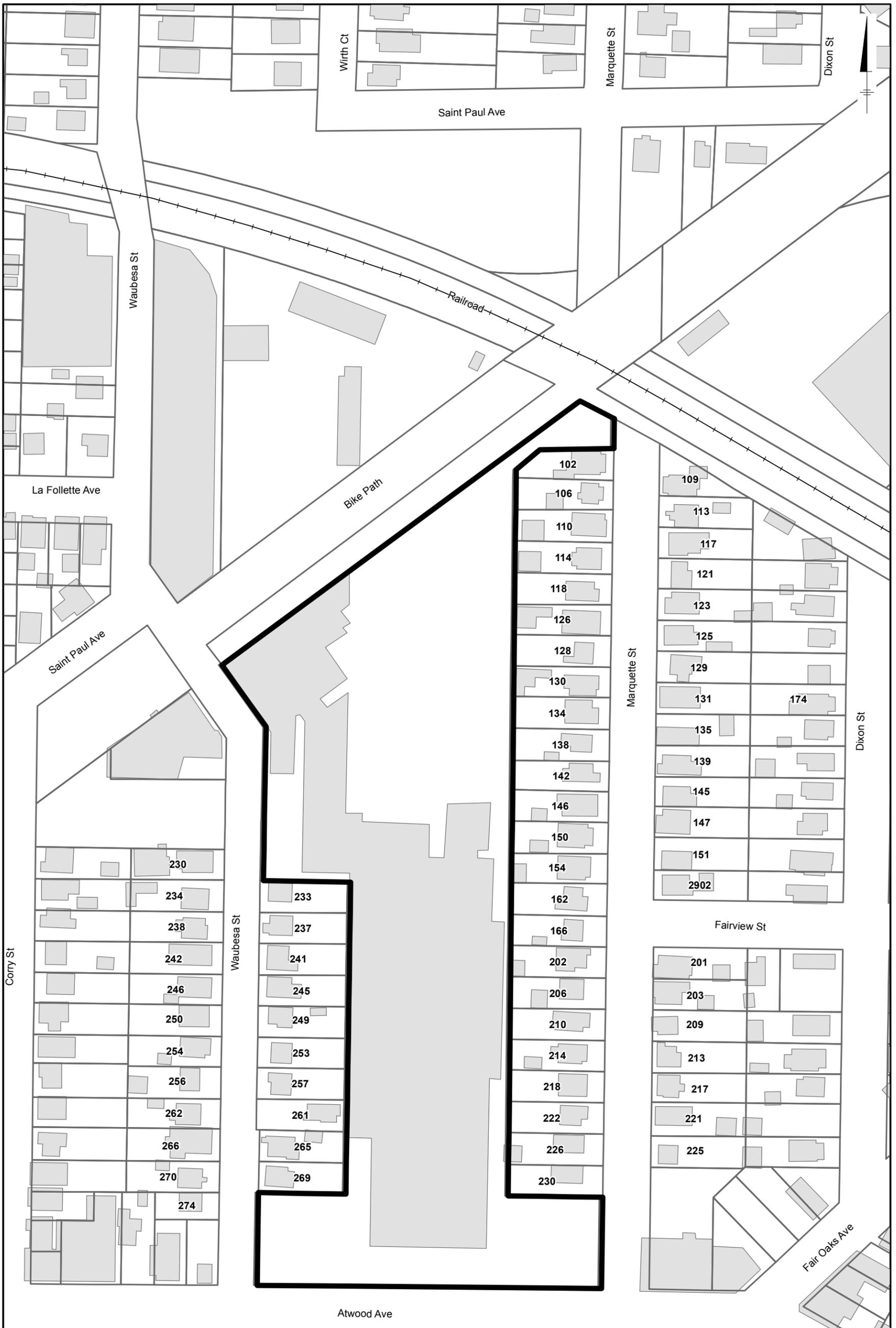
**SITE LOCATION MAP**



FIGURE

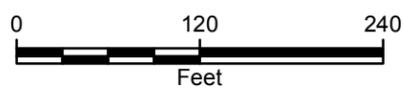
1-1

CITY: MPLS\_DIV/GROUP: IM DB: MG LD: CK MADISON-KIPP  
Z:\GISPROJECTS\ENR\MadisonKipp\Map2015-05\Fig1\_2\_SITE\_LAYOUT\_20150511.mxd



**LEGEND**

-  PARCELS
-  BUILDING FOOTPRINTS
-  SITE



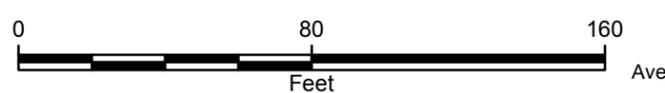
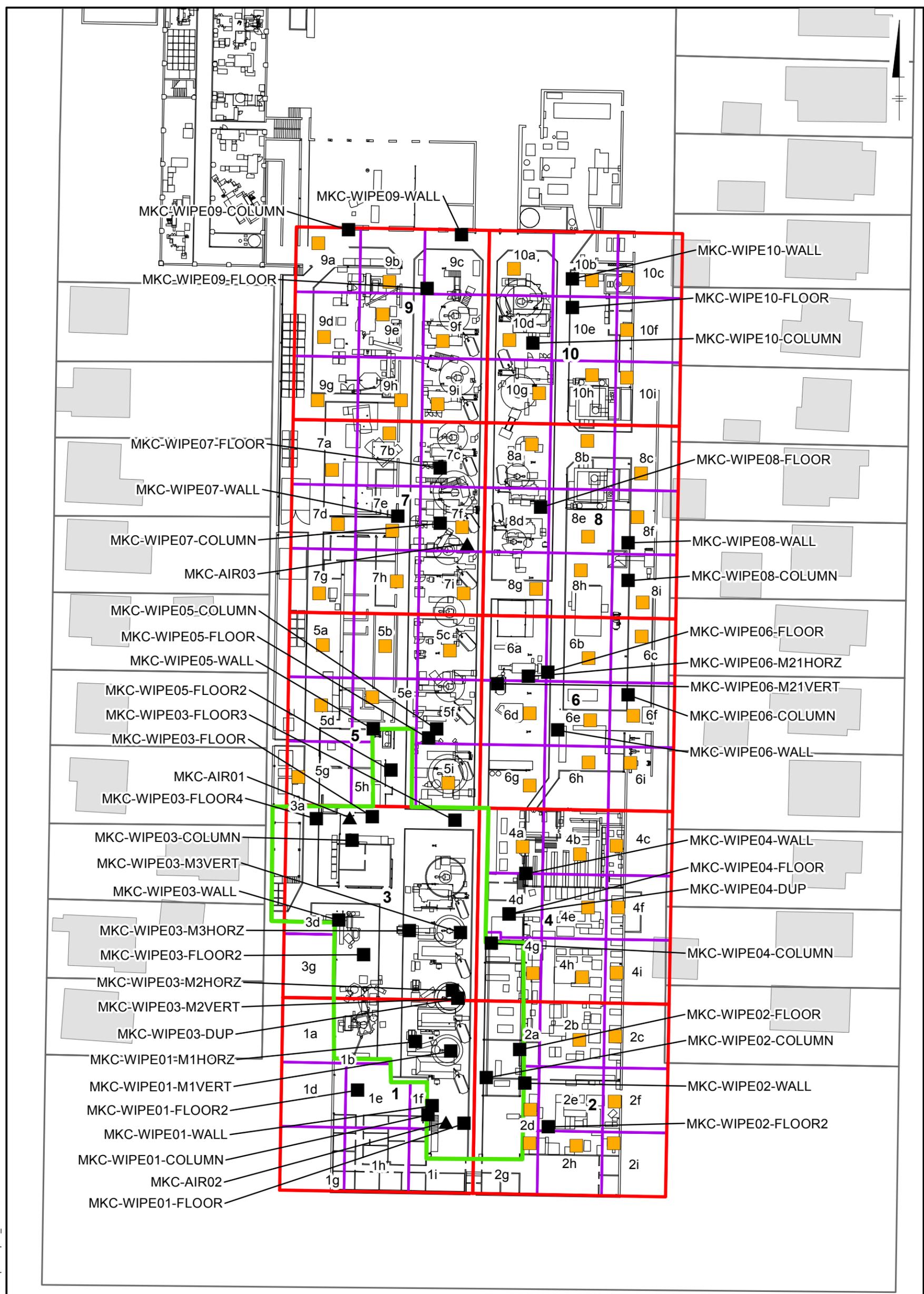
MADISON-KIPP CORPORATION  
201 WAUBESA STREET  
MADISON, WISCONSIN  
**WORK PLAN**

**SITE LAYOUT MAP**



FIGURE  
**1-2**

CITY: MKE DIV/GRUP: IM\_DB: GM\_LD: CK MADISON-KIPP  
 Z:\GIS\PROJECTS\ENV\MadisonKipp\Map\2015-06\Proposed\_InteriorWipeSamples\_20150611.mxd

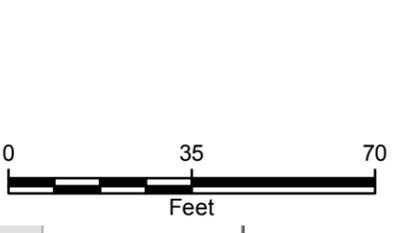
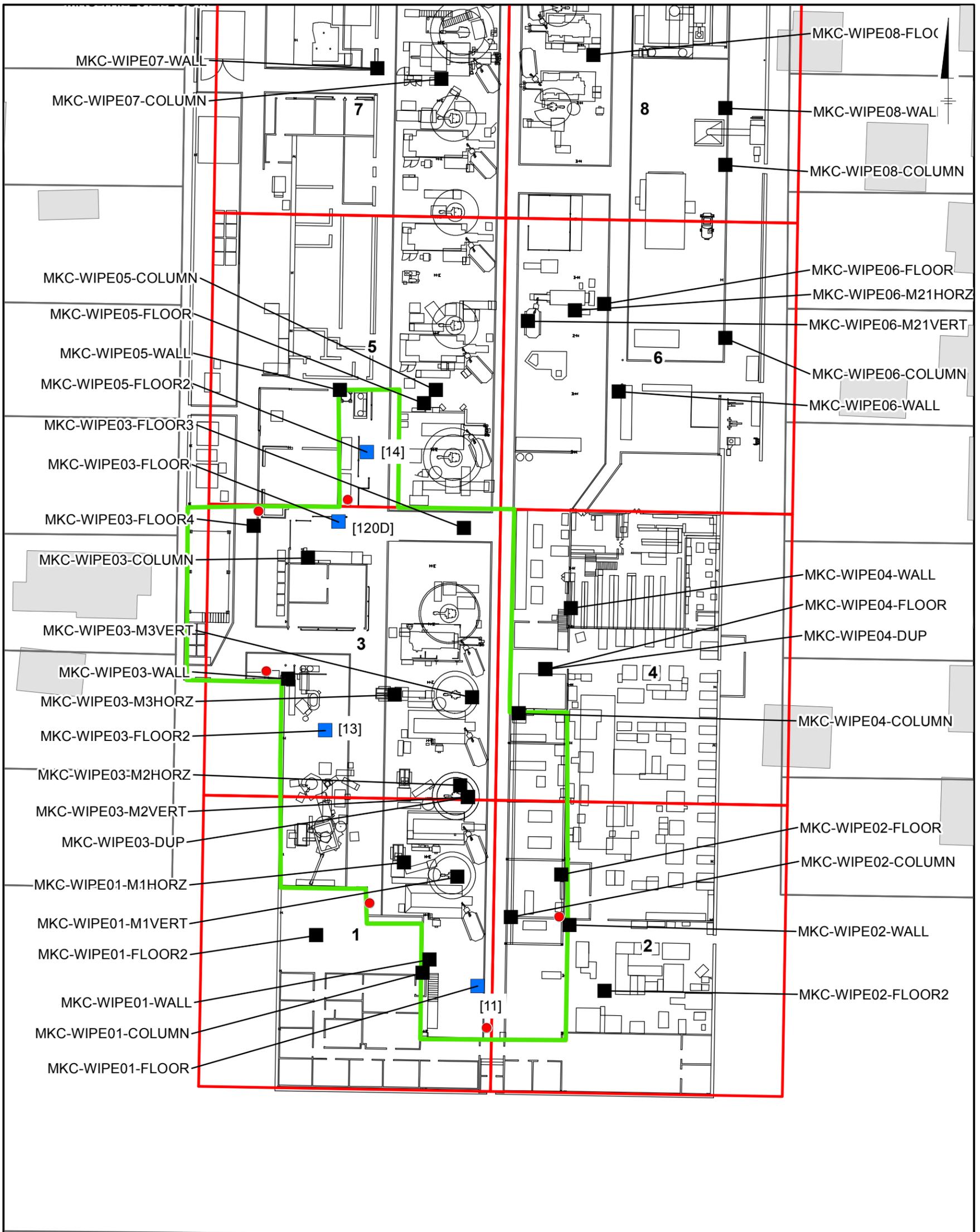


LEGEND	
	PREVIOUS INDOOR AMBIENT AIR SAMPLING LOCATION
	PREVIOUS WIPE SAMPLING LOCATION
	APPROXIMATE LOCATION OF FLOOR CONCENTRATIONS ABOVE CRITERIA
	100' GRID 1 100' GRID SQUARE ID
	33.3' GRID 2a 33.3' GRID SQUARE ID
	PROPOSED WIPE SAMPLING LOCATION (ACTUAL LOCATIONS OF WIPE SAMPLES WILL BE DETERMINED IN THE FIELD BASED ON VISUAL OBSERVATIONS)
	PLANT LAYOUT
	BUILDING FOOTPRINTS
	PARCELS

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 MADISON, WISCONSIN

**WORK PLAN**  
**PROPOSED INTERIOR BUILDING WIPE AND INDOOR AIR SAMPLING LOCATIONS**





CITY: MKE DIV/GRUOP: IM\_DB: GM\_LD: CK MADISON-KIPP Z:\GIS\PROJECTS\ENV\MadisonKipp\Map2015-06\Interior\_Wipe\_Locs\_Above\_20150608.mxd

LEGEND	
<span style="color: red;">●</span>	PCB MARKER LOCATIONS
<span style="color: blue;">■</span>	TOTAL DETECTED PCB CONCENTRATION REPORTED ABOVE US EPA PCB SPILL CLEANUP POLICY OF 10 µg/WIPE
<span style="color: black;">■</span>	TOTAL DETECTED PCB CONCENTRATION REPORTED BELOW US EPA PCB SPILL CLEANUP POLICY OF 10 µg/WIPE
<span style="border: 2px solid green; display: inline-block; width: 15px; height: 10px;"></span>	APPROXIMATE LOCATION OF FLOOR CONCENTRATIONS ABOVE CRITERIA
<span style="border: 1px solid red; display: inline-block; width: 15px; height: 10px;"></span>	100' GRID
	PLANT LAYOUT
	BUILDING FOOTPRINTS
	PARCELS
<b>1</b>	GRID SQUARE ID
[120]	CONCENTRATION IN µg/WIPE

MADISON-KIPP CORPORATION  
201 WAUBESA STREET  
MADISON, WISCONSIN

**WORK PLAN**

**APPROXIMATE LOCATION OF FLOOR CONCENTRATIONS ABOVE CRITERIA**

FIGURE  
**5-1**



## **Appendix A**

Environmental  
Chemistry Consulting  
Services PCB and  
Pesticide Wipe  
Analysis Supplemental  
Sample Collection  
Guidance



## **PCB AND PESTICIDE WIPE ANALYSIS Supplemental Sample Collection Guidance**

*Disclaimer: This document is not meant to replace 40CFR761 Subpart G – PCB Spill Cleanup Policy, or any other relevant Federal, state, or local requirements for collecting wipe samples. The guidance provided herein is for the sole purpose of communicating minimum requirements to collect a representative wipe sample for ultimate analysis by ECCS Method LAM-005 8082 PCBs or ECCS Method LAM-003 Organochlorine Pesticides. Attachment A is guidance from 40CFR relevant to PCB wipes and is provided herein for additional guidance on the subject topic.*

### **1.0 Purpose**

The purpose of this information is to provide guidance for the collection of wipe samples of solid surfaces potentially contaminated with polychlorinated biphenyl (PCB) and pesticide residues. Wipe samples are taken to determine the degree of surface contamination and provide information on environmental exposure.

### **2.0 Supplies Provided by Laboratory**

- 2.1 3 inch by 3 inch sterile gauze pads
- 2.2 4 ounce amber sample jars
- 2.3 80/20 iso-octane/acetone or hexane
- 2.4 5 ¾ inch Pasteur pipettes and bulb

### **3.0 Collection of Wipe Sample**

- 3.1 Select surface to be sampled. It should be a relatively flat and smooth surface that can be easily and thoroughly wiped with the gauze. A single sample will be of a 100 cm<sup>2</sup> area (10 cm x 10 cm). Identify the 100 cm<sup>2</sup> area(s) to be sampled in accordance with applicable regulations and guidance.
- 3.2 Put on a fresh clean pair of gloves for each wipe sample.
- 3.3 Prepare a gauze pad by moistening it with 80/20 iso-octane/acetone using a Pasteur pipette (do not soak).
- 3.4 Wipe the 100 cm<sup>2</sup> area in accordance with applicable regulations and guidance.
- 3.5 Be careful not to allow the gauze to touch any other surface, fold it over with the wipe surface to the inside and insert it into the sample jar in which the pad was shipped.
- 3.6 Repeat the above steps for additional samples.



**Attachment A**  
**EPA Guidance on PCB Wipe Sampling**

WIPE SAMPLING AND DOUBLE WASH/RINSE CLEANUP  
AS RECOMMENDED BY  
THE ENVIRONMENTAL PROTECTION AGENCY PCB SPILL CLEANUP POLICY

June 23, 1987

Revised and Clarified on April 18, 1991

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Chemical Regulation Branch  
United States Environmental Protection Agency  
Washington, D.C.

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  - d. Summary of Cleanup Levels Based on the EPA PCB Spill Cleanup Policy.
    - i. Low Concentration Spills Involving Less Than One Pound of PCBs by Weight.
      - ii. High Concentration Spills and Low Concentration Spills Involving More Than One Pound of PCBs by Weight.
  - e. Additional Wipe Sampling Information
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## I. WIPE SAMPLING ACCORDING TO THE PCB SPILL CLEANUP POLICY

### Introduction:

This document was prepared following the publication of the PCB Spill Cleanup Policy in the Federal Register on April 2, 1987. The procedures were demonstrated by EPA PCB program technical staff at PCB Forum '87 and PCB Forum '88. These PCB forums were privately sponsored seminars discussing the requirements of the recently issued PCB Spill Cleanup Policy. The seminars were publicly announced and held in eight cities near the EPA Regional Offices.

The revisions and clarifications to the document include the addition of an Introduction heading, the addition of three paragraphs to the Background heading, and the amendment to item 4 in "An Example of a Wipe Sampling Procedure."

This document was revised and clarified because it did not clearly and completely state EPA's intentions in an area where details were essential, that is the original version of this document assumed that a gloved hand would apply the gauze with moderate pressure, but inadvertently this requirement was never explicitly stated in the example of the wipe sampling procedure. The gloved-hand application of the gauze might have been assumed since the gloves were to be discarded after each sample. The procedure clearly did not say to apply the gauze to the surface with forceps. The EPA demonstrations and discussions at the PCB Forums clearly emphasized the pressurized application of moistened cotton gauze to the surface with a gloved hand.

### Background:

The PCB spill Cleanup Policy requires wipe sampling for the determination of surface levels of PCBs resulting from PCB spills onto hard, "smooth", surfaces such as metal, wood, concrete, plastic, and glass (see Tables 1 and 2). There are several activities surrounding a PCB spill cleanup where wipe sampling may be used: (a) site characterization; (b) interim evaluation of the progress of the cleanup; and (c) the final process to verify that the cleanup has met requirements of the PCB Spill Cleanup Policy.

Wipe sampling has a number of advantages. The most apparent advantage is that wipe sampling is probably the best way to determine smooth "impervious" surface concentrations. Wipe sampling is most effective in areas with relatively large, flat, easily accessible surfaces where an accidental and/or short time

exposure to PCBs has occurred. The surfaces which are sampled by wipe sampling in many cases will have been (or will be) cleaned by wiping or wiping-related activities.

Wipe sampling is best used in conjunction with statistical random sampling and/or area sampling techniques. Reduction in sampling errors for all kinds of sampling procedures can be accomplished by statistical selection of the smaller sampling sites selected to represent a larger area. Non-sampling errors may be reduced by maintaining consistency within the sampling activities; use of comprehensive quality control procedures and samples; and wherever possible, establishing a reference point for comparison.

Unfortunately, wipe sampling is not quantitative because of the fairly large variability in several component parts of sampling and the relative inefficiency of extraction of the analyte of interest from the wipes. Wipe sampling evaluation study results are known to vary widely, for example, when the same sampling is done (1) by different samplers; (2) on similarly contaminated surfaces having different textures or porosities; (3) using no solvent or solvents having different polarities; and (4) using different kinds of wiping material such as filter paper or cotton gauze.

When a decision is made to use wipe sampling, (1) it should be assumed that the results are not always reproducible; (2) extra care should be used to minimize the variability and optimize quantitation; and (3) even if representative sampling is employed, wipe sampling results can indicate residual levels substantially below true surface levels. In developing the PCB Spill Cleanup Policy, EPA has considered the advantages and disadvantages of wipe sampling and accordingly has established allowable residual PCB levels as measured by wipe sampling.

Since the objective of surface sampling is to remove PCB liquids and particles, which may be adhering to the surface, from the surface an aggressive sampling procedure is necessary. The aggressive sampling is appropriate since often the surfaces being sampled have been aggressively cleaned and may drive residual PCBs into the surface. For determining the PCB surface concentrations on smooth surfaces, EPA recommends wipe sampling using cotton gauze as the wipe medium and using a gloved or doubly gloved hand to apply the wipe to the surface. This procedure requires changing into new/clean gloves between samples. EPA recognizes that there may be some transport of PCBs from the gauze to the surface of the gloves. However, this potential loss is considered more acceptable than the problems from the disadvantages of other wipe sampling procedures.

Procedures employing filter paper and/or glass fiber pads and application of these pads to surfaces by swabbing, dipping, or brushing with a pair of forceps are unacceptable. EPA

recognizes that this kind of wipe sampling technique may be

widely applied to address other kinds of surface sampling objectives. However, to meet EPA's PCB surface sampling objectives, these procedures are less efficient and less effective than hand wiping with the more absorbent cotton gauze.

Any compositing of wipe samples or sampling of areas larger than 100 cm<sup>2</sup> may not address the intent of PCB Spill Cleanup Policy verification sampling.

### Answers to Questions on Wipe Sampling Procedures:

#### **Why is does it take so much care to wipe sample correctly?**

There is a considerable variability possible among wipe sampling results due to (a) the sampling technique of the sampler and (b) the efficiencies of removing PCBs from several matrices and placing the PCBs into several other matrices. Therefore it is important to reduce this variability to the maximum extent possible, so that in the event of a verification analysis by quality control samplers or government enforcement inspectors, similar wipe sampling results will be obtained for a clean site.

Two factors increase the probability of reducing errors introduced by the sampler's technique: consistency and quality control. Consistency is aided by proper training, easily understood sampling procedures, immediate availability of proper supplies, and whenever possible, using the same sampler to do all sampling at a particular site. Quality control procedures provide reference points and comparisons for the field sample results. When the analytical results from quality control samples indicate potential sampling and analysis problems, there is often sufficient time to reexamine field results. Quality control sampling can reduce or eliminate additional sampling and analysis start up and/or additional cleanup costs.

The reproducibility and efficiency of transferring residual PCBs from one place to another require that such residual PCBs must have a much greater affinity to partition, in one or more steps, from the place of origin to the ultimate destination. For all transfer steps, PCBs must exhibit a much greater propensity to be in the destination medium than in the medium of origin. There are several transfer steps in the process which starts from the removal of PCBs from the surface sampled and ends with the production of a PCB surface concentration by way of instrumental analysis.

The first of these transfer steps is removing residual PCBs from the surface to be sampled and transferring them into the sampling medium\*. Gauze pads are sturdier, allow better surface to surface contact, and absorb more solvent (and more PCBs) than filter paper. Therefore, gauze pads are the absorbent/sampling medium of choice. Since PCBs are very soluble in organic solvents, organic solvent is used to moisten the gauze pads to ease the transport of PCBs from the sampled surface into the sampling media. Once the areas of where the spill occurred have been sampled (after cleanup) and the residual PCBs have been transported to the moistened gauze, then the gauze is air dried and stored/shipped for chemical analysis. The gauze is dried so as to facilitate transfer by organic solvent from the gauze to another medium during the laboratory extraction step.

In the extraction step the PCBs must be isolated from the gauze in a form amenable to the chemical analysis methods to be used. The PCBs now in the gauze are usually extracted into a solvent by repeated rinsing with and subsequent collection of organic solvent. The extraction solvent is removed from the PCBs by evaporation of the solvent prior to chemical analysis. The more volatile organic solvent evaporates and leaves the less volatile PCBs in a more concentrated solution for further treatment or instrumental analysis.

**What is the best way to wipe sample for PCBs on smooth surfaces?**

There are several steps in a wipe sampling procedure. The first step is to prepare the sampler for the sampling activity. The sampler may have to be advised of (through a briefing or a refresher course), or trained in, the objectives of the sampling program and the procedures to be used to accomplish those objectives.

Once advised of the objectives and sampling procedures, the sampler must either prepare or obtain the sampling plan and sampling materials. The sampler must know the exact sampling sites or know the exact procedure for selecting those sites. The sampling supplies must be sufficient in quantity and quality for all normally expected occurrences. Provisions should be also made for quality assurance samples, chain of custody forms, and shipping materials for storage.

\* When PCB-contaminated office paper has been solvent rinsed, then wipe sampled and bulk sampled, some recent chemical analysis results indicate that the PCB concentration in the surface wipes is not the same as the concentration in the bulk samples. PCB levels in uncontaminated paper were used as a control. The difference in PCB levels in the wipe samples and bulk samples may

be explained by PCB migration into the paper either during cleanup to remove PCBs or during the wipe sampling step.

An important series of quality assurance measures taken before on-site sampling occurs may save considerable expense from collecting contaminated or unusable wipe samples. Sampler training can include practice sampling of surfaces spiked with PCB surrogate compounds, such as tri- and tetrachlorobenzenes to sharpen skills (a) in wiping thoroughly and consistently, and (b) avoiding cross contamination. In addition, before field sampling is conducted, method blanks can be used to verify that sampling equipment supplies and procedures do not introduce PCBs or analytical interferences to the wipe samples. Complete supplies for sampling should be cleaned, a fraction of the supplies sampled individually or through method blanks, and, if clean, the supplies should be protected against contamination or destruction while being transported to the sampling site and while at the sampling site before actual sampling occurs.

The sampler arrives at a sampling site and determines the exact location where the 100 square centimeter (cm<sup>2</sup>) sample will be taken. The sample location may be marked or framed by a template. The sampler must be conscious of possibility of cross contamination during all stages of the sampling activity. All surfaces should be wiped with as uniform a pressure as possible. It is important to use the appropriate pressure to thoroughly wipe materials off the surface. Wiping proceeds from left to right in rows from the top to the bottom of the framed sampling area. The sampling area is wiped again with the same uniform pressure in columns from the top to the bottom from the left side to the right side of the entire framed area. It is not critical whether wiping starts at the top left or with rows first and then columns. The objective is to systematically, thoroughly, and consistently wipe the entire framed area twice, each time from a different direction and orientation.

Once the area has been wiped, the sampling gauze is allowed to air dry and is replaced in the sample vial. The sample vial is then labelled, the chain of custody filled out, and the sample prepared/stored for shipping.

Table 1

**SUMMARY OF CLEANUP LEVELS  
BASED ON THE EPA PCB SPILL CLEANUP POLICY**

**Requirements for Cleanup of Low-Concentration Spills  
Which Involve Less Than One Pound PCBs by Weight  
(Less Than 270 Gallons of Untested Mineral Oil  
[Containing Less Than 500 ppm PCBs])**

Solid Surfaces (except for all indoor, residential surfaces other than vault areas)	Double washed/rinsed
All Indoor, Residential Surfaces Other Than Vault Areas	10 micrograms per 100 cm <sup>2</sup> by standard commercial wipe tests
Soil	Remove visible traces of the spill and soil within a one foot buffer of the visible traces

Table 2

**SUMMARY OF CLEANUP LEVELS  
BASED ON THE EPA PCB SPILL CLEANUP POLICY**

**Requirements for Cleanup of  
High-Concentration Spills and Low-Concentration Spills  
Involving One Pound or More PCBs by Weight  
(270 Gallons or More of Untested Mineral Oil  
[Containing Less Than 500 ppm PCBs])**

Residential/Commercial/Rural

Indoor (except vaults), and Outdoor High Contact	10 micrograms per 100 cm <sup>2</sup>
Indoor Vaults	10 micrograms per 100 cm <sup>2</sup>
Outdoor Low Contact Porous Surface Option	10 micrograms per 100 cm <sup>2</sup> 100 micrograms per 100 cm <sup>2</sup> plus encapsulation
Soil	10 ppm Plus a 10 Inch Cap

Restricted Access (Non-Sub-Station)

High Contact Surfaces	10 micrograms per 100 cm <sup>2</sup>
Low Contact Indoor Surfaces Porous Surface Option	10 micrograms per 100 cm <sup>2</sup> 100 micrograms per 100 cm <sup>2</sup> Plus Encapsulation
Outdoor Low Contact Surfaces	100 micrograms per 100 cm <sup>2</sup>
Soil	25 ppm

Outdoor Electrical Substations

Surfaces	100 micrograms per 100 cm <sup>2</sup>
----------	----------------------------------------

Soil

25 ppm or 50 ppm with Notice

**Additional Wipe Sampling Information  
(Contents)**

1. An Example of a List of Wipe Sampling Supplies.
2. An Example of Sample Site Preparations.
3. An Example of a Wipe Sampling Procedure.
4. A Detailed Description of Quality Controls for Wipe Sampling Activities.
5. Wipe Sampling Quality Control Samples (Summary).
6. An Example of Quality Assurance Procedures Useful When Conducting Wipe Sampling Activities.
7. An Example of Procedures to Use When Cleaning Wipe Sampling Equipment.

**An Example of a List of Wipe Sampling Supplies**

Copy of Sampling Procedures and Study Objectives  
Pen (Indelible Ink)  
Pre-numbered Sample Labels  
Tape to Cover Labels  
Chain of Custody Forms  
Screw Top Vials with Teflon Lined Caps  
    These Vials Contain Pre-Cleaned 3" x 3" Surgical Gauze Pads  
Teflon Squirt Bottle for Applying Solvent to Wipes and Washing  
Solvent, preferably in a bottle with a volumetric delivery top  
Graduated cylinder, when not using a volumetric delivery top  
Disposable Gloves  
Metal Ruler  
Sampling Template  
Forceps for Removing (Replacing) Gauze from (into) Vials  
Disposable Wipes (for cleaning ruler)  
Garbage Bags/Containers (for disposal of gloves and solid waste)  
Funnel  
Five Gallon Solvent Can for Disposal of Rinse Solvent  
Shipping/Storage Containers for Samples  
Sampling Site Description Forms with Optional Instant Print  
    Camera

### An Example of Sample Site Preparations

At each sample site location:

- Mark the exact sample site with the template or a ruler
  
- If the site is not easily marked with a template or ruler (an irregular non-planar surface), write a detailed description of the area sampled. A instant print photograph with the ruler included (for scale) is a very valuable descriptor.
  
- Prepare all necessary forms and sampling logs for entry of the sampling time, date, location, and other information describing the sampling at that particular site.
  
- Prepare all sampling equipment for sampling the site.

### An Example of a Wipe Sampling Procedure

Assume that the exact sampling site has been marked.

1. With gloved hands, remove the cap from the sampling vial.
2. With the forceps, remove the gauze from the sampling vial.
3. From a solvent bottle, use the volumetric delivery device or fill a graduated cylinder with 5 milliliters of solvent to the gauze.
4. Immediately begin applying the gauze using a gloved hand and, applying pressure, wipe the marked area completely twice, from left to right and then from top to bottom.
5. Let the gauze air dry.
6. Fold the dry gauze (sampled side inward) and return it to the sample vial.
7. Cap the sample vial.
8. Remove and discard the gloves.
9. Label the vial and fill out sampling details on the sampling forms.
10. Fill out chain of custody forms and prepare the sample for storage and shipping.

### **A Detailed Description of Quality Controls for Wipe Sampling Activities**

Several kinds of quality control (QC) samples should be used. Each kind of sample provides an indication of the reliability of a part of the sampling and analysis process.

It is better not to identify QC samples as such when submitting the QC samples to the analytical laboratory. It is best to randomly number all samples when submitting them to the analytical laboratory. The chemical analysis laboratory does not need to know sample descriptions except for matrix type or in the event of the presence of an unusually high concentration in the wipe. Specific identification of the QC samples will not be necessary since the concentration range in these samples should be in the normal operating range of the analytical instruments.

Vials refer to the glass vials containing sampling gauze.

1. Field Blanks - at least 5% of the total samples include at least two samples each from the following:
  - a. Ship unopened vials back for analysis.
  - b. With gloved hands, remove the cap from a sample vial for the estimated time (record this time) of normal wipe sampling, allow the gauze to air dry without applying it to any surface, and proceed with step 7 in the wipe sampling procedure.
  - c. Use the wipe sampling procedures to wipe some areas/surfaces near the sampling site but which are not expected to be contaminated.
2. Duplicates - at least 5% of total samples including at a minimum the designated samples from both the following groups:
  - a. Double wipe at least two sample sites, label which was the first wipe and which was the second wipe for each of the two sites, for each kind of surface sampled.
  - b. For at least two sample sites for each kind of surface sampled, wipe two adjacent identical or nearly identical areas. Clearly identify the samples as being adjacent to one another in the sample description forms.

**A Detailed Description of  
Quality Controls for Wipe Sampling Activities  
(Continued)**

3. Field Spikes - at least 5% of total samples including at a minimum the designated samples from each of the following groups for each kind of surface sampled. Clearly describe these samples on the sample description forms.
  - a. For two vials or more, remove each gauze and moisten as for sampling and spike each wet gauze with ten micrograms each of the kind of PCBs which was spilled, wipe a contaminated surface adjacent to a sampled surface as in 2b (above), let the gauze air dry, replace the gauze, and proceed with step 7 in the wipe sampling procedure.
  - b. For a second pair of vials or more, remove each gauze and moisten as for sampling, wipe a contaminated surface adjacent to a sampled surface as in 2b (above), after wipe sampling (but before air drying) spike each wet gauze with ten micrograms each of the kind of PCBs which was spilled, let the gauze air dry, replace the gauze in the vials, and proceed with step 7 in the wipe sampling procedure.
  - c. For a third pair of vials or more, spike sampling surfaces adjacent to another sampled surface as in 2b (above) with ten micrograms each of the kind of PCBs which was spilled and allow to air dry; remove each gauze and moisten as for sampling; wipe the surface; let the gauze air dry, replace the gauze in the vials; and proceed with step 7 in the wipe sampling procedure.

**Wipe Sampling Quality Control Samples (Summary)**

1. Field Blanks - At least two samples from each category
  - a. For each spill site prepare the following blanks:
    - i. Unopened sampling vials containing gauze
    - ii. Remove gauze but do not use to wipe
  - b. For each kind of surface, wipe an uncontaminated 100 cm<sup>2</sup> surface with a gauze as a blank surface
2. Duplicate Samples - At least 5% of total samples
  - a. For each kind of surface at each spill site:
    - i. Double wipe at least two sample sites
    - ii. Side by side wipe at least two sample sites
3. Spiked Samples - At least 5% of total samples
  - a. Wipe no less than two samples each for each kind of surface at each spill site. All are side by side paired samples. One sample for each pair is untreated, for the other sample:
    - i. Spike gauze with 10 micrograms of PCBs, then wipe the 100 cm<sup>2</sup> area
    - ii. Wipe the 100 cm<sup>2</sup> area first, then spike gauze with 10 micrograms of PCBs
    - iii. Spike the 100 cm<sup>2</sup> site with 10 micrograms of PCBs, then wipe

**An Example of Quality Assurance Procedures  
Useful When Conducting Wipe Sampling Activities**

1. Designate a person, not the sampler or chemical analyst, who is responsible for quality assurance and quality control including: training, preparation of sampling supplies, wipe sampling, sample preparation/extraction, chemical analysis, analytical data reduction, reporting of the sampling results, and conclusions drawn from the results.
2. Document the objectives of the wipe sampling and subsequent chemical analysis. Include performance requirements such as number of samples required, precision, accuracy, measurable deliverables, and schedules.
3. Develop a quality assurance plan which includes: the objectives; quality assurance/quality control procedures, audits, and schedules; persons responsible for all aspects of the sampling and chemical analysis efforts; references to all safety, training, sampling, and chemical analysis procedures; and corrective actions (including approximate times before corrective actions will occur) to be taken in the event that documented procedures cannot be or have not been followed.
4. Verify that staff doing sampling are the designated staff or suitably trained and informed replacements for the designated staff.
5. Verify that the sampling equipment and the sample gauze/vials are not going to introduce contamination into the samples.
6. Verify that sufficient quality control samples are taken and taken properly, that sampling objectives are met, and that chain of custody procedures are being followed.
7. Verify that sample extraction and chemical analysis occurs according to documented procedures. Assure that suitable and sufficient analytical quality control samples and reference standards are analyzed.
8. Verify that analytical data calculations are properly generated and the data are correctly associated with the proper samples.
9. Assure that conclusions based on the chemical analysis of the samples are in keeping with the sampling procedures and sample site locations.
10. Document quality assurance activities including: who did it, what was done, when it was done, where was it done, and why was it

done. Document and justify any deviations from documented procedures and policies.

**An Example of  
Procedures to Use When Cleaning Wipe Sampling Equipment**

1. Using clean (or cleaned) disposable equipment is overall probably more cost-effective than cleaning and verifying that cleaned sampling equipment is free from PCBs. The second choice is not cleaning any equipment on or near the sampling site, but to have sufficient recleaned sampling equipment to completely sample a site. The least favorable situation is to clean sampling equipment for reuse at the same sampling site. If cleaning must be done at or near the sampling site, clean the sampling equipment as far from the actual site of cleanup/contaminations as possible.

2. Try to have sufficient clean materials on-site to completely sample a site (plus at least ten percent surplus for unforeseen accidents and blunders) so as not to have to clean any sampling equipment.

3. Use cleaning procedures which have been verified as effective previously. Good cleaning includes:

- Washing with soapy water
- Rinsing thoroughly with water
- Rinsing three times thoroughly with distilled water
- Rinsing with PCB-free organic solvent
- Air drying for non-glass
- Drying in a muffle furnace at 350°C for glass
- Verification sampling and analysis of cleaned equipment
- Protective packaging for shipment to the sampling site

4. The same kind of verification procedures should be used for new equipment as is used for equipment which has been cleaned:

a. Selecting a statistical sample from the equipment. For lots having large numbers of units (such as sample bottles), a 5% or less proportion of the units may be sufficient. For equipment which comes in direct contact with contaminated surfaces (such as templates) a 10% sample may be more appropriate unless historical data have verified that a smaller proportion is sufficient.

b. Rinsing "clean", dry equipment with the same amount of organic solvent as is used in the sampling procedure or more than sufficient solvent to completely cover and rinse off all contact (with the wipe sample, sampler, or the surface) surfaces of equipment. The rinseate is collected and treated as an extract from a sample gauze pad.

c. The presence of detectable levels of PCBs indicate that

contamination is present and that the lot from which the verification sample(s) came must be either recleaned and reverified or disposed of appropriately.

## II. DESCRIPTION OF DOUBLE WASH/RINSE

### Introduction

The PCB Spill Cleanup Policy requires that low concentration spills of small amounts of PCBs on surfaces are to be removed by a double wash/rinse procedure. The objectives of the double wash/rinse are (1) to recognize the lesser hazard resulting from these small quantity spills and from the cleanup of such spills, and (2) to remove the easily removable PCB material thoroughly and quickly. It is also important not to redistribute PCBs or leave pieces of cleanup materials as a result of the cleanup procedure.

### General Requirements for All Double Wash/Rinse Surfaces

For spills where there is still visible PCB-containing liquid present on the surface to be cleaned up, the double wash/rinse procedure first requires a pre-cleaning step. This step includes thoroughly wiping/mopping up the entire surface with absorbent paper or cloth material, such that there are no longer visible signs of the liquid present on the surface.

The double wash/rinse procedure called for in the cleanup of surfaces contaminated by small spills includes the two washing steps and two rinsing steps. The two washing and rinsing steps are slightly different depending on: (a) whether a contaminated surface was relatively clean before the spill, or (b) whether a surface was coated/covered with some sort of absorbent material, such as dust, dirt, grime, or grease.

Minimization of residual PCBs following the double wash/rinse procedure is facilitated by the proper selection and use of cleanup equipment. Scrubbers and the absorbent pads used in the double wash/rinse procedure shall not be dissolved by solvents or cleaners used. Scrubbers and absorbent pads shall not contain greater than 2 parts per million (weight per weight) PCBs. Washing scrubbers and absorbent pads shall not be reused. Rinsing scrubbers and absorbent pads may be reused as washing scrubbers or absorbent pads if necessary, but this is not recommended. All double wash/rinse cleaning/absorbent materials must remain intact (i.e. do not shred, crumble, or leave visible fragments on the surface) after the double wash/rinse operation.

During the double wash/rinse process, all washing and rinsing liquids/solvents must be contained, captured, and properly disposed of in accordance with local, state, and Federal regulations. Following use in the double wash/rinse process, all double wash/rinse equipment and absorbent materials must also be disposed

of in accordance with local state, and Federal regulations.

## Summary of The Double Wash/Rinse Procedure

### General

1. Use disposable cleaning materials which do not
  - dissolve or break apart
  - contain traces of PCBs.
2. Remove any visible PCB liquid before washing/rinsing.
3. Capture and contain washing/rinsing solutions.
4. Properly dispose of cleaning materials and solutions/liquids.

### Specific

1. For surfaces not covered with dirt, dust, grime, grease or other potential absorbent of PCBs:

WASH 1: Scrub with organic solvent and wipe up the solvent.

RINSE 1: Wipe surface with moistened pad, wipe up with dry pad.

WASH 2: Repeat WASH 1.

RINSE 2: Repeat RINSE 1.

2. For surfaces covered with dirt, dust, grime, grease or other potential absorbent of PCBs:

WASH 1: Scrub with detergent and water, dry.

RINSE 1: Rinse with water, wipe with wet adsorbent pad, dry.

WASH 2: Scrub with organic solvent and wipe up the solvent.

RINSE 2: Wipe surface with moistened pad, wipe up with dry pad.



Detailed Requirements for the Double Wash/Rinse

1. Specific requirements for surfaces that do not appear dusty or grimy before a spill, such as glass, automobile surfaces, newly poured concrete, and desk tops:

WASH 1.

If there is no visible liquid or after having removed the visible liquid, cover the entire surface with organic solvent in which PCBs are soluble to at least 5% by weight. Contain and collect any runoff solvent for disposal. Scrub rough surfaces with a scrub brush or disposable scrubbing pad. Add solvent such that the surface is always very wet for one minute per square foot. Wipe smooth surfaces with a solvent-soaked, disposable absorbent pad for one minute per square foot. Any surface less than one square foot shall also be washed for one minute. Wipe, mop, and/or sorb the solvent onto absorbent material until no visible traces of the solvent remain.

RINSE 1.

Wipe the surface with an absorbent pad soaked with the same organic solvent with a solvent-soaked, disposable absorbent pad for one minute per square foot. Any surface less than one square foot shall also be washed for one minute. Immediately wipe/sop up the solvent on the surface with a dry absorbent.

WASH 2.

Repeat WASH 1.

RINSE 2.

Repeat RINSE 1.

Detailed Requirements for the Double Wash/Rinse (Continued)

2. Specific requirements for dirty, dusty, grimy, or greasy surfaces or surfaces having surface coverings of some other kind of sorbant materials (where the spill probably largely sorbed onto the materials on the surface):

WASH 1.

If there is no visible liquid or after having removed the visible liquid, cover the entire surface with concentrated or industrial strength detergent or non-ionic surfactant solution. Contain and collect all cleaning solutions for proper disposal. Scrub rough surfaces with a scrub brush or scrubbing pad, adding cleaning solution such that the surface is always very wet, for one minute per square foot. Wipe smooth surfaces with a cleaning solution-soaked disposable absorbent pad for one minute per square foot. Any surface less than one square foot shall also be washed for one minute. Mop up or absorb the residual cleaner solution and suds with an absorbent pad until the surface appears dry. This cleaning should remove any residual dirt, dust, grime, or other sorbant materials left on the surface following step one (above).

RINSE 1.

Rinse off the wash solution with one gallon of water per square foot and capture the rinse water. Mop up the wet surface until the surface appears dry.

WASH 2.

Next, cover the entire dry surface with organic solvent in which PCBs are soluble to at least 5% by weight. Scrub rough surfaces with a scrub brush or scrubbing pad adding solvent such that the surface is always very wet for one minute per square foot. Wipe smooth surfaces with a solvent-soaked, disposable absorbent pad for one minute per square foot. Any surface less than one square foot shall also be washed for one minute. Wipe, mop, and/or sorb the solvent onto absorbent material until no visible traces of the solvent remain.

RINSE 2.

Wipe the surface with an absorbent pad soaked with the

same organic solvent as in RINSE 1 (above) and immediately wipe up the solvent on the surface with a dry absorbent.



## **Appendix B**

Pace Analytical  
Services, Inc.  
Standard Operating  
Procedures (S-NY-O-  
241-rev.05 and S-NY-  
O-341-rev.02)



**STANDARD OPERATING PROCEDURE**

**EXTRACTION AND EXTRACT PREPARATION OF POLYURETHANE FOAM AIR CARTRIDGES FOR PCB ANALYSIS IN AIR CASSETTE MEDIA**

**Reference Methods: EPA Method TO-10A**

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SOP Number:	S-NY-O-241-rev.05
Effective Date:	03/03/15
Supersedes:	S-NY-O-241-rev.04

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**APPROVALS**

	03/03/15
_____ Assistant General Manager	_____ Date

	03/03/15
_____ Quality Manager	_____ Date

**PERIODIC REVIEW**

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date

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## 1. Purpose/Identification of Method

1.1. This is a Standard Operating Procedure for the extraction and cleanup of low volume polyurethane foam (PUF) air cassette samples for polychlorinated biphenyl (PCB) analysis using the Soxhlet extraction technique (Modified SW-846 Method 3540C/EPA Method TO-10A for subsequent analysis by SW-846 Method 8082A or EPA Method 680- PCB Homologs by GC/MS).

## 2. Summary of Method

- 2.1. Set up a Soxhlet extractor apparatus for each sample.
- 2.2. Load the PUF into the Soxhlet. Add the necessary surrogates and/or matrix spikes.
- 2.3. Load the extraction apparatus into its heating mantle and condenser and allow it to extract for  $18 \pm 2$  hours.
- 2.4. Solvent exchange and set to volume.
- 2.5. Samples are put through a series of cleanup steps, including sulfuric acid, Florisil slurry, and/or mercury shake. The samples are then submitted for gas chromatography (GC) analysis.

## 3. Scope and Application

- 3.1. **Personnel:** The policies and procedures contained in this SOP are applicable to all personnel involved in the extraction process for PUFs for PCB analysis.
- 3.2. The following procedure is utilized by Pace Analytical Services, Inc. for the extraction and cleanup of PCBs from PUF (air) samples using the Soxhlet extraction method of TO-10A PUFs for subsequent analysis by SW-846 Method 8082 or EPA Method 680.
- 3.3. **Parameters:** See method SOP for analyte list.

## 4. Applicable Matrices

- 4.1. This test method is appropriate for air samples sampled with TO-10A PUF media.

## 5. Limits of Detection and Quantitation

- 5.1. Please see determinative method SOPs (S-NY-O-148, EPA Method 8082A or Lab SOP, S-NY-O-040, EPA Method 680) for details.

## 6. Interferences

- 6.1. Solvents, reagents, and glassware may yield artifacts that interfere with sample analysis. All solvents and reagents are tested prior to use. Glassware must be cleaned as per SOP, S-NY-O-256. Method blanks are analyzed to demonstrate that the system is free of interferences.
- 6.2. Laboratory contaminants including phthalate esters may be introduced during extraction and subsequent cleanup procedures. The extraction technician should exercise caution that scrupulously cleaned glassware is used and that plastic tubing and other plastic materials do not contact samples or extracts.

6.3. The sample matrix itself is also a potential source for method analyte interference. The clean-up procedures provided in this SOP can be used to overcome many of these interferences.

## 7. Sample Collection, Preservation, Shipment and Storage

7.1. Samples are collected as per EPA method TO-10A, the client's Field Sampling, and Analysis Plan. Pace Analytical does not provide field sample collection services for air monitoring projects. Samples should be stored at  $>0-6^{\circ}\text{C}$  until shipping to laboratory.

7.2. Field samples are shipped to the laboratory in a cooler chilled with ice ( $>0-6^{\circ}\text{C}$ ).

7.3. Upon receipt samples are stored in laboratory under refrigeration at  $>0-6^{\circ}\text{C}$  until extraction.

7.4. Samples must be extracted within 7 days of collection and analysis must be performed within 40 days of extraction.

## 8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **Method Blank**- A method blank is processed with each batch of samples that are extracted to assess for contamination during prep and processing steps. The method blank is carried throughout all stages of sample preparation and extraction steps. A PUF is processed as the method blank.

8.3. **Laboratory Control Sample**- A non-site sample which is prepared in the laboratory, to which a known amount of target analyte is added for assessment of laboratory performance. The laboratory control spike sample is composed of a PUF with the spiked target analytes. The Laboratory Control Sample is processed with each batch of samples extracted.

8.4. **Laboratory Control Sample Duplicate**: An exact copy of the Laboratory Control Sample to further assess analyte recovery efficiency.

8.5. **Matrix Spike**: A site sample to which a known amount of target analyte is added for assessment of analyte recovery efficiency.

8.6. **Matrix Spike Duplicate**: An exact copy of the Matrix Spike utilizing the same site sample and known amount of target analyte for assessment of analyte recovery efficiency.

8.7. **Surrogate Compound Spike**: In chemical composition and chromatography similar to the analytes of interest. Usually not found in environmental samples. These compounds are spiked into all samples, standards, blanks, and matrix spike samples prior to analysis. Percent recoveries are calculated for each surrogate.

8.8. **QC-Quality Control**: A set of measures for each sample within an analysis methodology to assure that the process is in control.

## 9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Water Cooled Condenser: Pyrex 45/50 #3840-MCO (or equivalent).

9.2. 250mL Round Bottom Flask: Pyrex #4100 (or equivalent).

9.3. Soxhlet Repetitive Flushing (reflux) Unit: 45/50 Pyrex #3740-M (or equivalent).

- 9.4. Heating Mantle: Type "VF" laboratory heating mantle #HM0250VF1 (or equivalent).
- 9.5. Heating Mantle Controller: Glass-Col #PL3122 Minitwin (or equivalent) regulates temperature control of the mantle.
- 9.6. Chiller: Pump driven water circulating cooling system cool flow #75 NESLABS Instruments, Inc. (or equivalent).
- 9.7. Turbo Vap Evaporator: Zymark #ZW640-3.
- 9.8. Turbo Vap Evaporator concentrator tubes: Zymark 250mL, 0.5mL endpoint.
- 9.9. Beakers: Assorted Pyrex: 250mL, 600mL, and 1000mL, used for liquid containment and pipette storage.
- 9.10. Vials: glass, 4 dram and 40mL (with Polyseal sealed cap), for sample extracts.
- 9.11. Vial Rack: Plastic rack used to hold vials, during all phases of the extract processing.
- 9.12. Centrifuge: International Equipment Co., Model CL (or equivalent).
- 9.13. Wrist Shaker: Burrell wrist action shaker, Model 75 and 88 (or equivalent).
- 9.14. Pipettes: S/P Disposable Serological Borosilicate Pipettes:
  - 9.14.1. 1mL X 1/10 #P4650-11X.
  - 9.14.2. 5mL X 1/10 #P4650-15.
  - 9.14.3. 10mL X 1/10 #P4650-110.
  - 9.14.4. Kimble Pasteur Borosilicate glass pipette 9" #72050 (or equivalent).
- 9.15. Tweezers: Laboratory stainless steel tweezers used to place PUFs into Soxhlets and to squeeze extracted solvent out of PUFs into Soxhlets.
- 9.16. Replacement PUFs: ORBO-1000 or equivalent 22mm O.D. X 7.6cm length, pre-cleaned and pre-tested certified by Vendor. Supelco CAT# 20600-U.
- 9.17. Boiling Chips: Hengar #5785 Alltech Associates, Inc. (or equivalent).

## 10. Reagents and Standards

- 10.1. Hexane: High Purity Solvent Baxter (Burdick/Jackson) #UN1208 (or equivalent).
- 10.2. Diethyl Ether: Nanograde Mallinckrodt #3434-08.
- 10.3. Hexane/Ether: 95%hexane/5%ether for TO-10A by volume solvent mixture prepared in the lab. To prepare: Add 200mL of diethyl ether to 3800mL of hexane in a 4L bottle.
- 10.4. Florisil: J.T. Baker #M368-08. (or equivalent) Solvent washed per SOP S-NY-O-283.
- 10.5. Mercury: Triple distilled Mercury Refining Co, Albany, NY #328502 (or equivalent).
- 10.6. Sulfuric Acid: Na<sub>2</sub>SO<sub>4</sub> (concentrated) Mallinckrodt #2468 #UN1830 (or equivalent).
- 10.7. Surrogate Spike Solution: Laboratory prepared from primary stock solution Tetra-Chloro-meta-Xylene at 0.05ug/mL and Decachlorobiphenyl at 0.500ug/mL.
  - 10.7.1. To make 0.05ug/mL TCMX/0.5ug/mL DCBP in hexane: Allow a solution of 0.5ug/mL TCMX/5.0ug/mL DCBP in hexane to warm up to room temperature. Using a class A volumetric pipette, pipette 10.0mL of the solution into a 100mL volumetric flask. Fill the flask to 100mL with hexane. All information is recorded in the Standards Logbook.

10.8. Laboratory Control Sample Solution: Laboratory prepared from primary stock solution of PCB Aroclor at 1.00ug/mL.

10.8.1. To make a Spike Solution at 1.0ug/mL in hexane: Allow a solution of Aroclor at 100ug/mL in hexane to warm up to room temperature. Using a gas-tight syringe, measure out 1.0mL of the spike solution. Add it to a 100mL volumetric flask. Fill the flask to the 100mL mark with hexane. All information is recorded in the Standards Logbook.

## 11. Calibration and Standardization

11.1. Please see determinative method SOPs (S-NY-O-148, EPA Method 8082A or S-NY-O-040, EPA Method 680) for details.

## 12. Procedure

### 12.1. Sample Preparation:

12.1.1. Throughout the entire process it should be noted that if the technician encounters any problems or difficulties with any samples or steps involved, all work should **STOP!** Any problems should be brought to the attention of the supervisor and documented in the Laboratory Information Management System (LIMS).

12.1.2. Before any steps are taken, the technician should first review the sample job folder. The technician should also verify the sample IDs on the bottle against the chain of custody. If there is a discrepancy on either the sample label or the chain of custody, this should be documented in LIMS and brought to the attention of a supervisor.

12.1.3. Prior to extraction, all surfaces and fume hood used must be cleaned and wiped down with hexane. All work on PUF samples must be done in the designated "Low Level" hood.

12.1.4. PUF samples require all glassware to be pre-rinsed with hexane. PUF samples are for extremely low level PCB concentrations and require clean; hexane rinsed glassware.

12.1.5. Use extreme caution while using ether during this extraction. Ether and its vapors are extremely flammable and must be used in a fume.

### 12.2. Sample Extraction:

12.2.1. Rinse enough 250mL round bottoms and Soxhlets for each sample, blank, and QC sample. Place in fume hood and allow to dry.

12.2.2. After the glassware dries label them with a sample ID. To each round bottom, add a few boiling chips and approximately 200mL of 5% ether/hexane mixture.

12.2.3. Place a Soxhlet onto each round bottom, checking for cracks or chips that would cause solvent to leak out. Record the ID number of each Soxhlet and round bottom in LIMS.

12.2.4. Blank and QC PUFs are prepared using pre-cleaned replacement PUFs. For each sample, use a pair of tweezers to pull the PUF out of its PUF tube and push it into the appropriate Soxhlet. Depress both sides of the PUF and push the PUF to the bottom of the Soxhlet. Try to handle as PUF as little as possible. **Rinse tweezers with hexane in between handling each sample.**

12.2.5. Spike surrogate and spike compound solutions directly into the Soxhlet onto the PUF. The addition of spiking material to a sample, blank, or QC must be witnessed by another extraction technician.

Record the names of the technicians spiking and witnessing, surrogate and spike concentration, the amount spiked, and the spike solution reference code in LIMS.

12.2.6. Rinse the inside and the outside connecting joints of the condenser units that will be used with hexane. Turn on chiller to cool the condensers.

12.2.7. Place the round bottom flask with attached Soxhlet extractor onto a heating mantle and attach condenser unit. Turn corresponding thermostats on to setting 3.5. Double check Soxhlets at this time for any cracks or chips. Once the solvent begins to boil, a flushing action of three or more flushes per hour should be achieved.

12.2.8. The samples should be extracted for 18 hours  $\pm$  2 hours, usually overnight. Once the sample has finished extracting (usually in the morning), turn the heating mantle off and allow samples to cool to room temperature.

12.2.9. Once cool, disengage the Soxhlet from the condenser and move all round bottom/ Soxhlet units to a fume hood. The diethyl-ether in the samples will continue to release vapors. Using hexane pre-rinsed tweezers squeeze the extracted solvent from the PUF and into the Soxhlet. Tip the Soxhlet/round bottom unit to get the solvent in the soxhlet to drain into the round bottom.

12.2.10. Rinse Soxhlet with hexane and again tip to allow the unit to drain into the round bottom. Disconnect the Soxhlet from the round bottom and rinse the connecting joint of the Soxhlet into the round bottom. Set the Soxhlet aside at this time and leave it in the hood to dry.

12.2.11. Label turbo tubes with sample ID, one per sample, and record the ID of each Turbo Tube in LIMS. Rinse turbo tubes with hexane and allow to dry in the hood.

12.2.12. Add sodium sulfate to each round bottom, swirling contents. Add as much sodium sulfate as necessary until the drying agent is free flowing.

12.2.13. Pour the contents of the round bottom into the Turbo tube, decanting off the sodium sulfate. Rinse the last drop out of the mouth of the round bottom flask.

12.2.14. Add 3-4 Pasteur pipettes full of hexane to the round bottom. Swirl gently, and decant into same Turbo tube. Repeat twice more for each sample.

12.2.15. All glassware must be rinsed with acetone, and dried in the hood before being washed as per SOP S-NY-O-256.

### 12.3. Solvent Reduction: TurboVap Evaporator System:

12.3.1. The TurboVap evaporator system is used in place of the Kuderna Danish (KD)-concentrator apparatus. The TurboVap evaporator system is used to reduce the sample volume. The TurboVap uses a heated water bath and positive pressure nitrogen flow / vortex action. The unit maintains a slight equilibrium imbalance between the liquid and gaseous phase of the solvent extract, which allows fractional reduction of the solvents without loss of higher boiling point analytes.

12.3.2. Turn the unit on and allow to heat up to  $40^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .

12.3.3. As a precaution the TurboVap system regulators should be checked to assure that there is no residual gas pressure within the system and that the gas pressure regulator is off before placing samples in the apparatus. Residual gas pressure may cause splashing and cross contamination of samples. To bleed the system of residual gas pressure place an empty TurboTube into the water bath and close the lid. Ensure that the nitrogen gas pressure regulator is off. Bleed any residual gas until the regulator gauge reads "0" psi. Remove the empty TurboTube.

12.3.4. Wipe down inside of TurboVap with a hexane wetted paper towel including top lid and pins. Place TurboTubes containing the sample extracts into the TurboVap and close lid. Slowly open the

pressure regulator. Keep the gas pressure very low, until the solvent level is decreased, to avoid splashing. Increase the gas pressure as the sample reduces, maintaining uniform flow throughout the volume reduction.

12.3.5. The process for solvent (hexane/ether) reduction takes approximately 20-30 minutes. Do not leave the unit unattended as extracts may be blown to dryness and PCB loss may occur. Immediately notify a supervisor if an extract is blown to dryness.

12.3.6. Concentrate the solvent to approximately 2.5mL. Remove the samples from the TurboVap and place in a rack. If concentrating samples for 680 analysis, remove when the solvent is between 5mL and 10mL. Follow this SOP with the changes outlined in Attachment I.

12.3.7. Using a disposable Pasteur pipette, transfer the sample extract into a pre-rinsed volumetric. PUF samples are generally set to 5X, but client specifications may require a different set volume. Rinse Turbo Tube with two Pasteur pipette volumes of hexane, and transfer the rinse to the volumetric.

12.3.8. Add hexane to the volumetric meniscus mark. Decant the contents into a pre-labeled 4 dram vial.

#### 12.4. Sample Extract Cleanup:

12.4.1. Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection contain interfering substances which must be removed before accurate chromatographic analysis can be performed.

12.4.2. Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. The experience of the analyst combined with the sampling site history should guide the selection of which clean-up procedures are necessary. The sequence and number of repeats of cleanup steps performed are recorded by the sample preparation technician in LIMS.

##### 12.4.3. Sulfuric Acid Wash:

12.4.3.1. Sulfuric acid removes hydrocarbons and other organic compounds which are co-extracted with the PCB residues.

12.4.3.2. Add approximately 3.0mL concentrated H<sub>2</sub>SO<sub>4</sub> to vial and shake for 30 seconds by hand. Centrifuge for approximately 1 minute on a speed setting of ¾.

12.4.3.3. Transfer the hexane layer (top layer) to a new properly labeled 4 dram vial.

##### 12.4.4. Florisil Absorption (Slurry):

12.4.4.1. The Florisil slurry removes co-extracted polar compounds, residual water, and residual acid.

12.4.4.2. Add one spatula (approximately 0.5g) of tested and approved 10% Florisil to each extract vial (SOP S-NY-O-283 for how to prepare Florisil).

12.4.4.3. Vigorously shake the vial for approximately 30 seconds by hand.

12.4.4.4. Allow the Florisil to settle before proceeding on to the next step.

12.4.4.5. Transfer the hexane layer to a new properly labeled 4 dram vial.

##### 12.4.5. Elemental Sulfur Clean-up:

12.4.5.1. Elemental sulfur is soluble in the extract solvents used for sediment and soil samples. It is commonly found in many PUF/sediment/soil samples, decaying organic material, and some industrial wastes. Large amounts of sulfur can cause the electron capture detector (ECD) to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can

interfere with PCB measurement as a co-eluting chromatographic peak. PUF samples normally have less sulfur than sediment/soil samples.

12.4.5.2. Two techniques exist for the elimination of elemental sulfur in PCB extracts: mercuric precipitation (Mercury Shake) and the tetrabutylammonium (TBA) sulfite procedure. Tetrabutylammonium sulfite causes the least amount of degradation of a broad range of pesticides and organic compounds, while mercury may degrade organophosphorus and some organochlorine pesticides. The TBA procedure also has a higher capacity for samples containing high concentrations of elemental sulfur.

#### 12.4.6. Removal of Sulfur Using Mercury:

12.4.6.1. Add 1-3 drops of mercury to the sample extracts, cap, and place on the wrist shaker for 30 minutes. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract. A black precipitate may be seen in sample extracts containing elemental sulfur.

12.4.6.2. Transfer the extract a properly labeled 4 dram vial.

#### 12.5. Final Extract:

12.5.1. From the final extract vial, pipette approximately 1mL into a white screw top vial.

12.5.2. Complete all information in LIMS. Submit samples and project folder to the GC Analyst.

### 13. Quality Control

13.1. **Verification PUF sample:** A verification (a.k.a. certification) PUF sample is a cartridge assembly that is tested at the laboratory prior to delivery to field personnel. In general each vendor lot number is pre- tested by the laboratory.

13.1.1. Extract and prepare one pre-cleaned PUF cartridge assembly at a batch frequency described in the client's sampling/analytical plan (1 per 50 PUFs).

13.1.2. Submit extract for analysis by GC-ECD or GC/MS (EPA Method 8082A or EPA Method 680).

13.1.3. GC analysis of verification PUF must exhibit chromatogram free of PCB Aroclors (< Practical Quantitation Limit) and also be free of interfering non-target co-eluting contaminants.

#### 13.2. Laboratory Method Blank:

13.2.1. A laboratory method blank sample is prepared and extracted with each site sample extraction batch of up to 20 samples.

13.2.2. A pre-cleaned PUF is spiked with surrogate solution and extracted and prepared identically to project samples. The analyte concentration must be less than the Practical Quantitation Limit. If the blank concentration exceeds the PQL, the laboratory client is notified and data is qualified (B-flagged) and a case narrative is generated. All analysis must cease until the source of contamination is isolated and the problem is resolved. The default Practical Quantitation Limit for Method TO-10A is 0.100ug total PCB. Due to the nature of sample collection PUF samples cannot be re-extracted.

#### 13.3. Laboratory Control Samples (LCS/LCSD):

13.3.1. A laboratory control sample (LCS)/ laboratory control sample duplicate (LCSD) sample is prepared by spiking a pre-cleaned PUF cassette with an Aroclor of interest applicable to the project. The percent recovery must meet project specified or laboratory established limits. The default recovery limits are 70-130%.

13.3.2. Prepare LCS and LCSD samples at frequency specified in the clients sampling and analysis plan. The laboratory default is one LCS, LCSD per batch or 20 samples; whichever is greater. TO-10A PUFs are spiked at default with Aroclor mix in hexane at a concentration of 1.000ug/mL. Please see Table 23.1.

13.3.3. If the LCS/LCSD does not meet recovery limits, the extraction of samples must stop until the problem is identified and corrected. The client is notified and a case narrative is issued to the client along with the affected data describing the LCS failure. Re-extraction of PUF samples is not possible.

13.4. **Field Spike Sample:** A field spike sample is prepared for each 20 PUF cartridges supplied to field personnel or as the client's field sampling analysis plan requires. The spike is prepared in the same fashion as an LCS sample and is shipped to the field and then returned to the laboratory unopened. The field spike sample is extracted and analyzed with the sample batch. The percent recovery criteria and corrective action are the same as the LCS/LCSD sample described in Section 13.3. Please see Table 23.1 for spike amounts added to sample.

13.5. **Surrogate Spike:** Every site sample and QC sample is spiked with the TCMX/DCBP surrogate solution described in Table 23.1. The Surrogate recovery must meet project specified limits or default limits (60-120%). If the surrogate recovery does not meet specified limits then identify the problem, re-analyze extract by GC if necessary and provide a case narrative describing the problem along with associated sample concentration results. Please see Table 23.1 for surrogate spike amounts added to sample

13.6. **Field Blank Sample:** A field blank sample consists of a pre-cleaned cartridge assembly that is packaged and shipped to field personnel un-opened. The un-opened PUF is returned to the laboratory and analyzed with the sample batch. The PCB concentration should be less than the Practical Quantitation Limit (PQL). If PCBs are observed greater than the PQL compare results with laboratory method blank. Notify the client/field personnel of the problem and generate a case narrative that is issued with the analytical results. Due to the nature of sample collection, PUF samples cannot be re-extracted.

#### 14. Data Analysis and Calculations

14.1. See determinative method SOPs (Lab SOP S-NY-O-148, EPA Method 8082A or SOP S-NY-O-040, EPA Method 680) for details.

#### 15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See determinative method SOPs (Lab SOP, S-NY-O-148 EPA Method 8082A or SOP S-NY-O-040, EPA Method 680) for details.

#### 16. Corrective Actions for Out-of-Control Data

16.1. See determinative method SOPs (Lab SOP S-NY-O-148 EPA Method 8082A or SOP S-NY-O-040, EPA Method 680) for details.

#### 17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. See determinative method SOPs (Lab SOP, S-NY-O-148, EPA Method 8082A or SOP S-NY-O-040, EPA Method 680) for details.

## 18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. See determinative method SOPs (Lab SOP S-NY-O-148, EPA Method 8082A or SOP S-NY-O-040, EPA Method 680) for details.

## 19. Method Modifications

19.1. Not applicable to this SOP.

## 20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

## 21. Troubleshooting

21.1. Not applicable to this SOP.

## 22. Safety

22.1. The technician should have received in-house safety training and should know the location of first aid equipment and the emergency spill/clean-up equipment, before handling any apparatus or equipment.

22.2. Safety glasses, a lab coat, and gloves must be worn when handling glassware and samples.

22.3. Polychlorinated biphenyls have been tentatively classified as known or suspected carcinogens. The technician must review the Safety Data Sheets (SDS) for PCBs and all reagents used in the procedure before handling them. All equipment and solvents should be handled within a lab fume hood.

## 23. Waste Management

23.1. See SOP, S-NY-W-054 for details.

## 24. Pollution Prevention

24.1. See SOP S-NY-S-168.

## 25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. US-EPA SW-846 Test Methods for Solid Waste; Soxhlet Extraction Method 3540C; United States Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Vol.1B, Cincinnati, OH 45268. December 1996.

25.5. US-EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air Second Edition Compendium Method TO-10A. Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MS), 3/18/99.

25.6. Guide to Environmental Analytical Methods, Genium Publishing Corporation, Schenectady, NY 12304, 1997.

## 26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Table 1: Surrogate and Spike Additions.

26.2. Attachment I: Method Outline Summary.

26.3. Attachment II: Cleanup Procedures for EPA Method 680 with 1mL set volume.

## 27. Revisions

Document Number	Reason for Change	Date
S-NY-O-241-rev.5	General: converted to new format. Sections 25.1-25.3: added standard Pace references.	08Jan2015

**Table 1: Surrogate and Spike Additions**

<b>Fortification Mixture</b>	<b>Concentration</b>	<b>Volume added to Samples</b>
TCMX/DCBP Surrogate mix in hexane	0.05ug/mL TCMX/ 0.5 ug/mL DCBP	0.500mL
Aroclor 1016 Spike mix in hexane	1.000ug/mL	1.000mL
Aroclor 1221 Spike mix in hexane	1.000ug/mL	1.000mL
Aroclor 1232 Spike mix in hexane	1.000ug/mL	1.000mL
Aroclor 1242 Spike mix in hexane	1.000ug/mL	1.000mL
Aroclor 1248 Spike mix in hexane	1.000ug/mL	1.000mL
Aroclor 1254 Spike mix in hexane	1.000ug/mL	1.000mL
Aroclor 1260 Spike mix in hexane	1.000ug/mL	1.000mL

## **Attachment I: Method Outline Summary**

- Step 1:** PREPARE FUME HOOD AND SAMPLES FOR EXTRACTION
- Step 2:** RINSE GLASSWARE AND LET DRY
- Step 3:** SET UP SOXHLET EXTRACTOR APPARATUS
- Step 4:** ADD SURROGATES AND/OR MATRIX SPIKE
- Step 5:** EXTRACT SAMPLE FOR APPROXIMATELY 18 HOURS +/- 2hours
- Step 6:** BREAKDOWN SOXHLET EXTRACTOR APPARATUS
- Step 7:** TRANSFER SOLVENT TO TURBO TUBE
- Step 8:** SOLVENT REDUCTION, USING THE TURBOVAP EVAPORATION SYSTEM
- Step 9:** SET TO VOLUME
- Step 10:** EXTRACT CLEANUP (ACID, FLORISIL, MERCURY)
- Step 11:** GC SCREENING/ ANALYSIS

## **Attachment II: Cleanup Procedures for EPA Method 680 with 1mL set volume**

- Follow procedure steps from 14.0 through 14.3.6.
- Concentrate the samples to between 5mL and 10mL.
- Transfer the samples to a 40mL vial **without setting to volume**. Rinse the Turbo tube twice with hexane and transfer that rinse to the same vial. Set the Turbo tube aside and DO NOT rinse. It will be used again later in the process.
- Add approximately 3mL of Sulfuric Acid to each sample. Shake, and then centrifuge. Transfer the hexane layer to a new vial.
- Once the hexane has been transferred, back rinse the sulfuric acid layer with one Pasteur pipette of hexane. Transfer that rinse into the sample vial.
- Add Florisil and shake the sample, then allow the Florisil to settle. Transfer the hexane into a new vial.
- Back rinse the Florisil with a Pasteur pipette of hexane. Transfer that rinse to the sample vial.
- Add 2-3 drops of mercury to each sample, then shake for 30 minutes. Remove the samples from the wrist shaker and transfer the hexane to the original Turbo tube.
- Back rinse the mercury with one Pasteur pipette of hexane. Transfer the rinse to the turbo tube containing the sample.
- Place the Turbo tubes in the Turbo Vap and concentrate until the samples are at approximately 0.5mL.
- Remove from the Turbo Vap and set to volume using a hexane-rinsed class A 1mL volumetric flask. Once the sample is set to volume, pipette it from the volumetric flask and into an amber auto sampler vial. The sample is ready for analysis.



**STANDARD OPERATING PROCEDURE**

**DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)**

**IN T O4A/T O10A**

**Reference Methods: EPA TO4A and EPA TO10A**

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LOCAL SOP NUMBER:	S-NY-O-341-rev.02
EFFECTIVE DATE:	03/30/15
SUPERSEDES:	S-NY-O-341-rev.01

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**APPROVALS**

	03/30/15
_____ Assistant General Manager	_____ Date

	03/30/15
_____ Quality Manager	_____ Date

**PERIODIC REVIEW**

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date
_____ Signature	_____ Title	_____ Date

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## 1. Purpose/Identification of Method

1.1. This standard operating procedure (SOP) is used to determine polychlorinated biphenyl (PCB) Aroclors in ambient air by gas chromatography with electron capture detection and total Aroclor quantification using EPA Methods TO-4A and TO-10A

## 2. Summary of Method

2.1. Samples are extracted with a pesticide analytical grade solvent. The extracts are further processed by concentration and a series of clean-up procedures. The sample extracts are then analyzed by injecting onto a gas chromatographic system equipped with an electron capture detector.

2.2. The purpose of this SOP is to provide a detailed written document for quantification of PCBs as Aroclors according to EPA Methods TO-4A and TO-10A specification.

2.3. This SOP provides detailed instructions for gas chromatographic conditions, calibration, and analysis of PCBs as Aroclors by gas chromatography. Sample extraction and cleanup procedures are described separately in additional laboratory Standard Operating Procedures.

2.4. Extensive knowledge of this SOP and EPA Methods TO-4A and TO-10A is required. The analysis portion of this method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.

## 3. Scope and Application

3.1. **Personnel:** The policies and procedures contained in this SOP are applicable to all personnel involved in the analysis of PCBs by EPA Method TO10A and EPA Method TO4A.

3.2. **Parameters:** The following PCB Aroclors can be determined by this method:

<u>COMPOUND</u>	<u>CAS NUMBERS</u>
AROCLOR 1016	12674-11-2
AROCLOR 1221	11104-28-2
AROCLOR 1232	11141-16-5
AROCLOR 1242	53469-21-9
AROLCOR 1248	12672-29-6
AROLCOR 1254	11097-69-1
AROCLOR 1260	11096-82-5
AROCLOR 1262	37324-23-5
AROCLOR 1268	11100-14-4

## 4. Applicable Matrices

4.1. This SOP is applicable in the determination and quantification of PCBs as Aroclors as outlined in the EPA Method TO-10A and EPA Method TO-4A. It is applicable in air cassette samples including polyurethane foam (PUF).

## 5. Limits of Detection and Quantitation

5.1. The following are default reporting limits based on the lowest calibration standard:

Matrix	Sample Mass/Volume Extracted	Calibration Curve Low Standard	Extract Volume	Global MDL* (All Aroclors)	RL (PQL) (all Aroclors)
Polyurethane foam cassette (TO10A/TO4A)	1 Puf	20ng/mL	5mL	*	0.100ug/Puf

5.2. Individual MDLs are determined every two years for each instrument with matrix specific studies for each extraction methodology. MDLs must be determined again whenever a major change in instrumentation or extraction methodology occurs.

5.3. MDLs are verified annually by the extraction and analysis of a low level MDL verification check sample. The Aroclor must be observed qualitatively in the MDL verification check sample.

5.4. Global MDL values can be obtained by request from the QA department.

## 6. Interferences

6.1. Laboratory contamination can occur by the introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters exhibits response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification. Laboratory method blanks must be thoroughly reviewed for presence of non-target peaks and comparison of samples with blank chromatographic patterns.

6.2. Polychloroterphenls (PCT), polybrominatedbiphenyls (PBB), polychlorinated naphthalenes (PCN), as well as dioxins can co-elute with PCBs. Carry-over from these compounds, when in high concentrations, is common if clean-up procedures are not followed. These materials may be removed through the use of specified clean-up procedures.

6.3. Pesticides can be a source of contamination through breakdown into components such as hexachlorobenzene (HCB). This chlorinated compound can carry-over on GC column, and contaminate samples. Specified clean-up procedures should be followed to eliminate this as a source of contamination when analyzing PCBs. High concentrations of pesticides can cause carry-over on GC columns.

## 7. Sample Collection, Preservation, Shipment and Storage

### 7.1. Sample Collection and Preservation:

7.1.1. Project specific, the jars maybe required to be pre-cleaned to EPA specification protocol A – recommended for extractable organic, semivolatile and pesticide analysis. Protect samples from light.

7.1.2. All samples must be placed on ice or refrigerated at 4°C (± 2°C) from the time they are collected until delivery to the lab. Samples collected and delivered to the laboratory on the same day may not reach 4± 2°C. Sample cooling is considered adequate if samples are received on ice.

### 7.2. Sample Shipment:

7.2.1. Sample Shipment is accomplished through a carrier such as Federal Express or United Postal Service for overnight 1-day delivery to the lab. Shipment is normally handled by the field personnel collecting the samples and coordinated with sample receiving department at the lab. Samples can also be picked up by the lab courier service if samples are collected within driving distance to the lab.

### 7.3. Sample Storage:

7.3.1. The samples must be protected from light and refrigerated at  $>0-6^{\circ}\text{C}$  from time of receipt until they are removed from storage for extraction. Remaining sample material will be stored protected from light and refrigerated at  $>0-6^{\circ}\text{C}$ . Sample will be disposed of or stored / archived according to project specifications.

### 7.4. Sample Extract Storage:

7.4.1. Sample extracts must be protected from light and stored refrigerated at  $>0-6^{\circ}\text{C}$  during the analysis. After analysis is complete, sample extracts will be discarded after 60 days.

7.4.2. Field samples, sample extracts, and calibration standards must be stored separately.

### 7.5. Required Hold Time:

7.5.1. Extraction of PUF samples by appropriate technique must be completed within seven days from sample collection.

7.5.2. Sample extracts must be analyzed within one year of sample extraction.

## 8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. Accuracy- Accuracy means the nearness of a result or the mean ( $\pm$ ) of a set of results to the true value. Accuracy is assessed by analysis of reference samples and determination of percent recoveries.

8.3. Analytical Batch- The basic unit for analytical quality control is the analytical batch. The analytical batch is defined as samples, which are analyzed together whereas the sample method sequence, the reagent lots, and manipulations are common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition (e.g., ground water, sludge, ash, etc.).

8.4. Calibration Standard - A series of known standard solutions used by the analyst for instrument calibration. Calibration standards are prepared from primary standard and/or stock standard solutions.

8.5. CAS Number - An assigned number used to identify a chemical. CAS stands for Chemical Abstracts Service, an organization that indexes information published in Chemical Abstracts by the American Chemical Society and that provides index guides by which information about particular substances may be located in the abstracts. Sequentially assigned CAS numbers identify specific chemicals, except when followed by an asterisk (\*) which signifies a compound (often naturally occurring) of variable composition. The numbers have no chemical significance. The CAS number is a concise, unique means of material identification. (Chemical Abstracts Service, Division of American Chemical Society, Box 3012, Columbus, OH 43210: [614] 447-3600).

8.6. Continuous Calibration Verification Standard (CCS) - A calibration standard containing the method analyte and surrogate, which is analyzed periodically to verify the accuracy of the existing calibration for those analytes.

8.7. Duplicate - A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

- 8.8. Environmental Sample - An environmental sample or field sample is a representative sample of any material (aqueous, no aqueous, or multimedia) collected from any source for which determination of composition or contamination as requested or required. Environmental samples are normally classified as follows. Water/Wastewater--raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents.
- 8.9. Initial Calibration - Analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the analytical detector or method.
- 8.10. Instrument Calibration - Analysis of analytical standards for a series of different specified concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.
- 8.11. Laboratory Control Sample (LCS) - Also known as the Quality Control (QC) Check Standard or Quality Control (QC) Check Sample. The LCS consists of an aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added. The LCS is extracted and analyzed exactly like a field sample, and its purpose is to determine whether the analysis is in control and whether the laboratory is capable of making accurate and precise measurements.
- 8.12. Matrix - The predominant material of which the sample to be analyzed is composed of. Matrix is not synonymous with phase (liquid or solid).
- 8.13. Method Blank - A laboratory derived sample consisting of reagent water or other blank matrix that consists of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The laboratory method blank is used to define the level of laboratory analyte background or other interferences that exist in the laboratory environment, the reagents, or the apparatus.
- 8.14. SDS- Safety Data Sheet - OSHA has established guidelines for the descriptive data that should be concisely provided on a data sheet to serve as the basis for written hazard communication programs. The thrust of the law is to have those who make, distribute, and use hazardous materials responsible for effective communication. See the Hazard Communication Rule, 29 CFR, Part 1910, 1200, as amended, Sec. g. See Schedule I, Sec. 12, of the Canadian Hazardous Products Act.
- 8.15. Precision - Precision is the agreement between a set of replicate measurements without assumption of knowledge of the true value. Precision is assessed by means of duplicate/replicate sample analysis.
- 8.16. Quality Control- Set of measures within a sample analysis methodology to assure that the process is in control.
- 8.17. Standard Curve- A standard curve is a curve which plots concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by diluting the stock analyte solution in graduated amounts which cover the expected range of the samples being analyzed. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards must be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.
- 8.18. Stock Standard Solution - A concentrated standard solution containing the method analyte. This stock standard can be used to prepare other more dilute standards.
- 8.19. Surrogate Standard - A pure compound added to a sample in the laboratory prior to extraction so that the overall efficiency of a method can be assessed.
- 8.20. Surrogate Analyte - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are added to all laboratory method blanks, laboratory QC reference samples (Laboratory Control Spikes), laboratory duplicates, calibration and continuing check standards, field samples,

field duplicate samples, field matrix spike samples, field matrix spike duplicate samples prior to extraction and/or analysis. Percent recovery is calculated for each surrogate to assess extraction efficiency.

8.21. Primary Standard Solution - A solution of several analytes prepared from stock solutions that can be diluted as needed to prepare calibration solutions and to prepare other standard solutions.

## 9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Pace Analytical Services will use a Varian Model 3800 (or equivalent) gas chromatograph equipped with Model 1077 split/splitless injector (or equivalent), temperature programmable oven, LEAP RE PAL automatic sampler (or equivalent), and electron capture detector (or equivalent). A data system and integration of detector signal is interfaced to the gas chromatograph.

9.2. Chromatographic Data System: A data system for measuring peak height and peak area. An Empower computer network based workstation (Waters Corporation), will be employed to capture detector response and digitally store the chromatographic, electronic peak integration for precise calculations, database structuring of the analytical information, and archival capabilities.

9.3. Column (Primary Hydrogen Carrier Gas): ZB-1MS, Phenomenex Cat. No 7FD-G011-08; 20m x 0.18mm x 0.18um.

9.4. Column (Secondary Hydrogen Carrier Gas): ZB-5, Phenomenex Cat, No 7FD-G002-08; 20m x 0.18mm x 0.18um.

9.5. Class A volumetric flasks: 5.0-100mL.

9.6. 4 dram vials for sample extract storage.

9.7. Pasteur pipettes.

9.8. 250mL and 100mL beakers, glass.

9.9. Disposable 1.0, 5.0, and 10.0mL pipettes.

9.10. Hexane, Burdick and Jacksons-Pest Grade (or equivalent).

9.11. Acetone, Burdick and Jacksons-Pest Grade (or equivalent).

9.12. Toluene, Baker (or equivalent).

9.13. Methylene Chloride, Burdick and Jackson (or equivalent).

9.14. Ferrules: 0.4mm graphite/vespel, Restek 20229, and ¼" graphite ferrules, Restek 20210 (or equivalent).

9.15. Injectors septa: Thermolite Septa, Restek 20365 (or equivalent).

9.16. Injectors liner: Low Pressure Drop Liner w/Glass Wool, Restek 21033 (or equivalent).

9.17. SGE Injector Syringe 10.0uL: SGE 002987 or equivalent.

9.18. Auto Sampler vials: Snap vial 12x32mm Clear w/P, Microliter 11-5200 (or equivalent).

9.19. Snap Caps: 11mm Natural Snap Cap PTFE, Microliter 11-0051N-B (or equivalent).

## 10. Reagents and Standards

### 10.1. Aroclor Stock Standard Solutions:

10.1.1. Polychlorinated Biphenyls - Stock standards are prepared from individual Aroclor stock solutions from Accustandard. See Attachment 1 Table 1 for the exact preparation of each compound.

10.1.2. The stock standards are transferred into screw-cap boston bottles and stored in a freezer 0°C, protected from light. Stock standards should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced after one year, or sooner if a problem with instrument calibration is detected.

PCB Formulation	Supplier	Catalog #	Conc. (PPM)
A1016	Accustandard	C-216S-H-100x	10000.0
A1221	Accustandard	C-221S-H-100x	10000.0
A1232	Accustandard	C-232S-H-100x	10000.0
A1242	Accustandard	C-242S-H-100x	10000.0
A1248	Accustandard	C-248S-H-100x	10000.0
A1254	Accustandard	C-254S-H-100x	10000.0
A1260	Accustandard	C-260S-H-100x	10000.0
A1262	Accustandard	C-262S-H-10x	1000.0
A1268	Accustandard	C-268S-H-10x	1000.0
TCMX/DCBP (surrogate)	Ultra Scientific	CUS-4911	500/5000

\*unless otherwise noted hexane is the solution used to make all dilutions.

### 10.2. Calibration Standards:

10.2.1. Calibration standards are prepared at five concentration levels using a prepared working standard. See Attachment I, Table 2 and for the preparation and exact concentrations of the working standards. The following five standards make up the initial calibration curve standard set for a High Level curve: 20ng/mL, 100ng/mL, 250ng/mL, 500ng/mL, 1000ng/mL. The following five standards make up the initial calibration curve set for a Low Level curve: 5ng/mL, 10ng/mL, 20ng/mL, 50ng/mL, 100ng/mL.

10.2.2. The two surrogates Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in the A1254 calibration standards. The standard for TCMX/DCBP is prepared by diluting 1mL

of TCMX/DCBP custom standard solution (ULTRA, cat.#CUS-4911, at 500/5000 ng/mL) into a 1000mL volumetric flask resulting in a solution of TCMX/DCBP at 0.5/5.0ug/mL.

10.2.3. Refer to Attachment I, Table 3 for instructions on preparation of the calibration standards containing A1254 and the surrogates. Refer to Attachment 1, Table 2 for instructions on preparing the remaining calibration standards.

10.2.4. Transfer all calibration standards to ASE vials and store in a refrigerator at 0-6°C, protected from light. Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem.

### 10.3. PCB Continuing Calibration Stock Standards:

10.3.1. The stock standards are transferred into a screw cap boston bottles and stored in a refrigerator protected from light. Stock standard should be checked frequently for signs of evaporation, especially just prior to preparing calibration standards. Stock PCB standards must be replaced annually, or sooner if a problem with instrument calibration is detected.

<b>PCB</b>	<b>Supplier</b>	<b>Catalog #</b>	<b>Conc. (ug/mL)</b>
A1016	Chem Service	S-11086J	1000
A1221	Chem Service	S-11087J	1000
A1232	Chem Service	S-11088J	1000
A1242	Chem Service	S-11089J	1000
A1248	Chem Service	S-11090J	1000
A1254	Chem Service	S-11091J	1000
A1260	Chem Service	S-11092J	1000
A1262	Ultra Scientific	EPA-1372	1000
A1268	Ultra Scientific	EPA-1382	1000

### 10.4. Continuing Calibration Standards:

10.4.1. The surrogate compounds Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP) are included in all Continuing Calibration Check Standards at a concentration near the mid-point of the surrogate calibration curve sequence. All continuing calibration standards are prepared independently from calibration standards, by using an alternate source purchased from standard vendors. Refer to Attachment II, Tables 1-3 for instructions on preparation of these standards.

## 11. Calibration and Standardization

11.1. Gas chromatographic operation parameters: See Attachment III.

## 11.2. Initial GC Calibration:

11.2.1. GC calibration is performed by the external standard calibration procedure. Prior to running samples the system must be calibrated and system performance must be verified.

11.2.2. Establish the gas chromatographic operating parameters outlined in the Procedure section and prepare the calibration standards at the five concentrations outlined in the Reagent and Standard section. Inject each calibration standard using the GC Autosampler and the parameters outlined in the Procedure section. Note: The same parameters are used for actual samples.

11.2.3. For each Aroclor, 5 peaks are selected to prepare calibration curves. The peaks selected from the multi-component Aroclor formulations were based on maximizing the separation for each Aroclor (i.e., minimizing peak overlap in retention time). Consideration was also given to selecting peaks that normally did not have problems with co-elution with interfering peaks or possible co-elution with organochlorine pesticides. The determined area of the five peaks selected for calibration is processed by the data workstation as a group, combining the area for calculations of the calibration factors. The following table lists the Aroclors that are included in the initial calibration and the peak numbers used.

<u>Aroclor</u>	<u>Peak Numbers</u>
A1016	6, 7, 8, 9, 10
A1221	1, 2, 3, 4, 5
A1232	5, 7, 8, 9, 10
A1242	6, 7, 8, 9, 10
A1248	11, 12, 13, 14, 15
A1254	16, 17, 18, 19, 20
A1260	20, 21, 22, 23, 24
A1262	20, 21, 22, 23, 24
A1268	23, 24, 25, 26, 27

11.2.4. For the initial calibration curve to be considered valid, the percent relative standard deviation of response factors must be less than 20% over the working range if average calibration factor quantitation is used. Note: the % RSD is a useful check for linearity through the origin and is used as a data quality indicator. In general an inverse weighted linear calibration curve with intercept is used for quantitation and is not replaced with the average calibration factor. For linear calibration curve the Correlation Coefficient R must be greater than 0.990.

11.2.5. Once linear calibration has been established it is subjected to an additional check. This check is the comparison of the calculated amount of the low calibration standard for each Aroclor against the expected amount of the standard using the % difference. Re-fitting the calibration data back to the model or calculating the % difference is determined by using the following equation:

$$\% \text{ Difference} = (C_c - C_e / C_e) \times 100$$

Where  $C_c$  = Calculated amount of standard, in mass or concentration units.

$C_e$  = Expected amount of standard, in mass or concentration units.

The absolute value of the percent difference between these two amounts for

Every calibration level should be less than or equal to 20%.

11.2.6. Our laboratory uses a computer based chromatography software module (Water Corporation, Empower software) interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. Integration of peak areas and production of chromatograms is performed in the Empower software.

All data analysis will be carried out in Empower including calculating calibration curves/response factors, report generation, and archival of data.

11.2.7. If a re-calibration is performed, the CCCS must be analyzed again and values calculated using the new relative response factors. If the CCCS fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (*i.e.*, GC gas leak, autosampler syringe plugged, broken injector liner).

### 11.3. Retention Time Windows:

11.3.1. The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Select a calibration standard and inject three times over a 72-hour time period.

11.3.2. For each peak calculate the standard deviation resulting from the variation in the three retention times for that peak.

11.3.3. The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.

11.3.4. If the standard deviation of the selected peak is zero, then a default standard deviation of 0.01 minutes is used. If it is the last eluting peak that the zero for the standard deviation, then substitute the standard deviation of the peak eluting before the last peak.

## 12. Procedure

12.1. This analytical procedure is specifically performed for analysis of air samples. The following SOP's detail sample extraction procedures that are utilized in preparing samples for analysis by this analytical method.

SOP NAME	TITLE	EPA Method
S-NY-O-151	PUF Extraction for TO-4A analysis	3540C/ TO-4A
S-NY-O-241	PUF Extraction for TO-10A analysis	3540C/ TO-10A

### 12.2. Gas Chromatograph Procedures:

12.2.1. Prescreening of sample extracts: See standard operating procedure S-NY-O-140 for details on the PCB screening procedures used prior to final analysis by this method. Prescreening is a fast and effective way to determine if re-extracts are required and dilutions for over ranged samples. The GC will be standardized by using Aroclor 1221, Aroclor 1242, and Aroclor 1260. These three Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution for this determinative method. A three level calibration curve is utilized (0.50ug/mL, 2.5ug/mL, and 5.0ug/mL standards). The concentration of each Aroclor (grouped as Aroclor 1221, Aroclor 1242, and Aroclor 1260 only) in a sample will be calculated based on extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted for final capillary GC analysis. If a dilution is necessary, sample extracts are diluted to a solution concentration near 0.500ug/mL, so ensuring each sample quantifies in the middle of the calibration curve.

12.2.2. Approximately 1.0mL of the final dilution extract is then transferred into a labeled autosampler vial.

12.2.3. The sequence of the analytical queue is set up in the Laboratory Information Management System (LIMS) as a unique batch file. This file contains the exact order in which standards, instrument blanks, and samples will be analyzed. Once the sample set is uploaded into the Empower acquisition/run screen and saved, the sample set is printed and the samples are loaded into the GC autosampler tray in the order specified by the sample set queue.

12.2.4. The following labeling will be used on the autosampler vial and for the sample set file created for the analytical queue.

12.2.4.1. The initial calibration standard will be labeled as 040516A, 040516B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

12.2.4.2. The instrument blanks will be labeled 070405B01, B02, B03, etc. Substitute the actual date of analysis in the file name.

12.2.4.3. The continuing calibration check standards will be labeled CS160405A CS160405B, etc. Substitute the actual date of analysis and the Aroclor used in the file name.

12.2.4.4. Samples are labeled with the laboratory identification number on the autosampler vial. In the sample set file the laboratory identification number, along with the client identification, sample weight, set volume and dilution are entered.

12.2.5. At this point the chromatography software can be initiated to start data collection. The gas chromatograph is placed into run mode and sample analysis is performed until the analytical queue is complete.

12.2.6. Peak Identification:

12.2.6.1. Target peaks are identified in unknown samples based upon Retention Time (RT). The retention time of an unknown peak must fall within the retention time windows established.

12.2.6.2. Besides using retention time windows to assign peak IDs, the analyst should also rely on their own experience in recognition of multi-response PCB chromatograms. Caution should be exercised when identifying peaks which elute near interferences present in samples and blanks. Comparison of sample chromatograms with method blank and field blank chromatograms is useful in determining chromatographic interferences.

12.2.6.3. This method should be applied with caution when used in determining PCB of interest in unknown sample for which no prior historical information exists. In this case confirmatory column analysis or confirmation by GC/MS analysis may be advised.

12.3. Data Reduction/Reporting:

12.3.1. Final peak assignments and quantitation calculations are performed within the software along with the current instrument calibration. The final concentration results are provided in the reporting section of the software. Final concentration results are reviewed by QA department or other approved manager before release to the client.

12.3.2. Data Qualifiers: Sample Concentration Reports (Certificates of Analysis, Data Package Form 1's and Electronic Data Deliverables (EDDs) are generated using the appropriate data qualifiers as follows:

12.3.2.1. U- Denotes analyte not detected at concentration greater than or equal to the Practical Quantitation Limit (PQL). Note: PQLs are adjusted for sample weight/volume and dilution factors.

12.3.2.2. J- Denotes an estimated concentration. The concentration result is greater than or equal to the Method Detection Limit (MDL) but less than the Practical Quantitation Limit (PQL).

12.3.2.3. P- Indicates relative percent difference between primary and secondary GC column analysis exceeds 40%.

12.3.2.4. C- Denotes analyte confirmed by secondary GC column analysis.

12.3.2.5. B- Denotes analyte observed in associated method blank. Analyte concentration should be considered as estimated.

12.3.2.6. E- Denotes analyte concentration exceeded calibration range of instrument. Sample could not be re-analyzed at secondary dilution due to insufficient sample amount, quick turn-around request, sample matrix interference or hold time excursion. Concentration result should be considered as estimated.

12.3.2.7. Z- Laboratory Reserved Qualifier (explained in associated Case Narrative).

### 13. Quality Control

13.1. This Method Blank- With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps.

13.1.1. The method blank must exhibit PCB levels less than the matrix defined minimum detection limit (MDL)/reporting limit (RL). If the method blank exhibits PCB contamination above the reportable MDL/RL, the samples associated with the contaminated blank should be flagged with a "B" indicating blank contamination. The value measured in the blank is reported for those samples associated with the particular blank out of criteria.

13.1.2. Method blanks can be reported with a "B" flag if the analyte detected is less than 10% of the regulatory limit associated with an analyte or is less than 10% of the sample result for the same analyte, whichever is greater.

13.2. Laboratory Control Sample (LCS)-

13.2.1. A Laboratory Control Spike (LCS) and a Laboratory Control Spike Duplicate, also referred to as a QC reference check standard, is extracted with each batch of samples at a rate of one per 20 samples. For Puf samples spike 1 PCB free Puf, extract and analyze. An Aroclor is chosen for the LCS and LCSD, typically base on program requirements or expected sample contamination. Calculate the percent recovery for the PCB spike. If the percent recovery for the LCS or LCSD is out of criteria (70-130%) the analysis is out of control and the problem should be immediately corrected.

13.2.2. The following are default Laboratory Spikes Concentrations:

13.3.2.1. Puf Samples: 1.0mL of A1242 at 1.00ug/mL in hexane set to 5mL final extraction volume.

13.3.2.2. Note: Alternate spike concentrations and selection of Aroclors may be applicable based on project specific requirements.

13.3.3. A Laboratory Control Spike Duplicate (LCSD) – Duplicate analysis of the LCS is performed to assess method precision. The relative percent difference of the two measurements on the LCS and LCSD is calculated on total PCB concentration by the following equation:

$$RPD = (DUP1-DUP2)/AVG \times 100$$

RPD = Relative Percent Difference

DUP1 = The greater of the measured values

DUP2 = The lesser of the measured values

AVG – Average of the two analyses relative percent difference must be less than or equal to 30%.

13.4. Surrogates:

13.4.2. A surrogate compound is added to each sample, method blank, LCS, and LCSD at time of extraction. The surrogate compounds chosen for this method are Tetra-chloro-meta-xylene (TCMX) and Decachlorobiphenyl (DCBP). The following are typical surrogate amounts added to normal encountered matrices. These amounts can be adjusted if the PCB background levels are high and surrogate is being diluted out of analysis range.

13.4.2.1. Puf: 0.5mL of 0.05 ug/mL TCMX/0.50 ug/mL DCBP in hexane set to 5mL final extract volume.

13.4.3. Surrogate compound is added to all instrument blanks that are analyzed after CCCS. Surrogate compound must meet required limits of 60-120%.

13.4.4. The surrogate recoveries must fall within the require limits of 60-120%.. If percent recovery is not within the required limits for either surrogate, the following steps are required.

13.4.4.1. Review calculations that were used to generated surrogate percent recovery values to make certain there are no errors

13.4.4.2. Check by GC analysis surrogate solutions used during sample extraction steps to ensure that no problems exist with spiking solutions.

13.4.4.3. Review data for chromatographic interferences.

13.4.4.4. Re-analysis of samples may be indicated if problems persist with surrogate recoveries. If the surrogate percent recovery is out of limits re-analysis samples, low or high surrogate recovery is due to matrix affects and the data can be reported as estimated. If above steps do not lead to satisfactory results then consult with organics manager to resolve the situation.

13.5. Continuing Calibration Check Standard (CCCS):

13.5.2. The initial CCCS is from an alternative source independent of the calibration check standards. It is prepared at a concentration approximately equal to the midlevel calibration standard. This standard is analyzed after the initial calibration standards, every ninth injection, and at the end an analytical sequence. One check standards must be run within a 12 hour analytical shift. The percent recover must be +15 of the true value.

13.5.3. If the criterion is exceeded, the analyst should inspect the system to determine the cause and perform maintenance as necessary. The system can then be recalibrated and sample analysis can proceed. Note that all samples which are not bracketed by valid check standards must be re-analyzed when the system is in-control.

13.6. Retention Time:

13.6.2. The retention time (RT) windows are established from the continuing calibration check standard (CCCS) peak retention times. The CCCS is analyzed three times over a 72 hour period and the standard deviation is calculated from the three retention time measurements. The standard deviation is multiplied by three and this establishes the retention time window for each

quantified peak (+3SD). Use the retention time for a peak in the continuing calibration check standard to determine the midpoint of the retention time window for the analysis sequence. If the continuing calibration checks fall outside of these windows update the windows using the previous check standard. If the retention times are still outside the established windows the instrument maintenance must be performed and recalibration may be required.

- 13.6.3. This function is performed in the chromatography software graphically as vertical dropdown retention time markers with retention time window brackets. Besides using the retention time window to assign peaks for quantification, the analyst should also rely on their experience in pattern recognition of multi-response sample analysis.
- 13.6.4. Retention time studies are available upon request from the QA department.
- 13.7. Analytical Sequence Queue: The following is an example of the order that initial calibration standards, continuing calibration check standards, method blanks, QC samples, and samples are placed in an analytical sequence. A continuing calibration check standard is run after every nine samples in the analytical sequence. All analytical sequences must end with a continuing calibration check standard regardless of the number of samples. Below is an example of an analytical sequence.

Injections	Material Injected
1-2	Hexane Blank
3-47	Initial Calibration Standards
48	Hexane Blank
49-57	Continuing Calibration Check Standard
58	Hexane Blank w/surrogates
59-68	Samples analyses, including method blanks, matrix spikes, matrix duplicates, matrix spike duplicates, and QC reference check standard. A maximum of nine samples between continuing calibration check standards
69	Continuing calibration check standard
70	Hexane Blank w/surrogates
71	Repeat injections 59-68 sequence

- 13.8. PCB Aroclor Qualitative Identifications and Secondary GC column Confirmation:
  - 13.8.2. Positive identification of PCB Aroclors is based on comparison of retention time of the five selected quantitation peaks and major non-quantitation peaks for the unknown sample with retention time of reference standards (continuing calibration verification standards). Additionally pattern recognition is used for comparison of unknown samples with reference standards for positive identification.
    - 13.8.2.1. In cases where multiple Aroclors are present with overlapping chromatographic patterns or interferences are encountered that are not removed with extract cleanup processes one or two quantitation peaks may be dropped and not used for quantitation. A minimum of 3 quantitation peaks must be used for all unknown samples and standards. When quantitation peaks are dropped for a sample or standard the corresponding peaks are also dropped in the initial calibration sequence for calculation purposes.
  - 13.8.3. Confirmation of Aroclor presence by secondary GC column analysis may be necessary for highly altered/degraded PCB patterns or for programs including PCB air monitoring, US-EPA

CLP protocol and other projects as specified in the site sampling and analysis quality assurance plan.

- 13.8.4. Dual Column/Confirmatory Column Analysis by GC- Inject samples under same operating conditions and analytical run QA/QC parameters on a secondary GC column of dissimilar phase (e.g., ZB-1 and ZB-5). Note: If using dual GC column system, samples are injected sequentially through separate injection ports onto both columns. Samples are analyzed and concentration results are reported.
- 13.9. Dual Column/Confirmatory Column Laboratory Default by SW-846:
  - 13.9.2. Report lowest concentration of the 2 column results for each individual Aroclor on the merged EDD, Form 1 or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects)
  - 13.9.3. If RPD percent exceeds 40% report the lowest concentration results of the two analyses unless observed chromatographic interference or instrumental analysis QA/QC indicates the higher value may be more accurate. P-flag all excursions > 40% and describe interferences or rationale for reporting lower value in Data Narrative.
  - 13.9.4. If a concentration is above the PQL on one column and below PQL on the second column, the qualitative presence is not confirmed and the sample is reported as not detected. Note: If reporting to the MDL is required do the following: For reporting to the MDL: a) If one result is greater than the PQL and other result is <PQL (J-flag) report the highest result as confirmed (unless interference or QC reasons indicate value); b) If one result is above MDL (J-Flag) and second is Not Detected report the concentration as not detected. (Presence not confirmed); c) If both results are J-Flag values (<PQL) report the lowest value of the two.
- 13.10. USEPA-CLP/ASP Program Protocols:
  - 13.10.2. Report Lowest Value of the 2 column results for each individual Aroclor on the merged EDD, Form 1 or Certificate of Analysis (Note: This is appropriate for Aroclor regulated projects. E.g. Air Monitoring for EPA TO-10A alternative reporting may be based upon total PCB values for PCB-total regulated projects).
  - 13.10.3. If Percent Difference (not RPD%) exceeds 25% then P-flag all excursions >25%. Note any chromatographic interferences present in Case Narrative.
  - 13.10.4. If one results is greater than PQL and the results is < PQL (J-Flag) Report the lowest result (J-Flagged) value (confirmed hit).
  - 13.10.5. If one result is above MDL (J-Flag) and second is Not detected, report the concentration as not detected (presence not confirmed).

## 14. Data Analysis and Calculations

14.1. PCB Solution concentration calculation from initial Calibration by Linear Regression:

$$Y_i = aX_i + b$$

$X_i$  = Calibrated Solution Concentration (ng/mL)

$Y_i$  = total area response of 5 PCB quant. peaks (uV-Sec.)

a = slope

b = intercept

Note: In those instances where samples may be quantitated with 3-4 peaks due to interference or overlap, the Empower system automatically quantitates against the calibration using only the area of the selected peaks.

**Unknown Solution Conc.  $X = (Y - b) / a$**

Y = Total area response of PCB Chromatogram (uV-Sec.)

a = slope of ICAL by linear regression

b = intercept of ICAL by linear regression

#### 14.2. Capillary GC: Sample calculations:

14.2.1. The concentration of each identified PCB Aroclor in a sample will be calculated based on the sample weight or volume.

14.2.2. The PCB solution concentration of the extract is calculated as follows:

**Solution Conc. =  $(Y - b) / a$**

Y = Total area response of PCB Chromatogram (uV-Sec.)

a = slope of ICAL by linear regression

b = intercept of ICAL by linear regression

14.3. Final concentration of samples- Calculations of final PCB concentrations will vary upon matrix, calculations for PUFs are as follows:

#### **PUF Cassette:**

**Final Concentration =  $(\text{Sol. Conc.}) * (V) * \text{DF} / V_a$  ng/cubicmeter**

Sol Conc. = Solution Concentration (ng/mL)

V = concentrated extract volume (mL)

DF = analytical dilution factor

V<sub>a</sub> = volume of air sampled (cubicmeter)

The calculated concentration for each PCB Aroclor will be compared to its respective sample-specific reporting limit (RL) and method detection limit (MDL). The results with concentration at or above the MDL but below RL will be reported as detects and flagged as estimated J. The results for peaks with concentrations at or above the RL would be reported as unqualified numeric values

### 15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. The GC analyst is responsible for generating the data and also is the initial individual to review the data. This would include inspection of the chromatographic data, processing the raw data, producing all required data forms, inspection of calibration curves for compliance, surrogate recovery, laboratory control spike recovery, matrix spike/matrix spike duplicate recovery, and continuing calibration compliance.

15.2. Once the initial review of the data is performed by the analyst, decisions are made at that time to accept the data if all criteria are met or to reject sample data if any of the quality control parameters or limits are out of control. Depending on the situation, samples requiring re-extraction will be notified to the appropriate extraction personnel, sample extracts requiring re-injection will be queued for analysis, new calibrations may have to be performed, or samples re-analyzed due to failing continuing check standards.

15.3. The analyst may also consult with the Quality Manager as to the best form of action to take or if the situation warrants corrective action beyond routine practices. If no recourse is available and the data is to be reported out of criteria, a Case Narrative Report is generated and the deviation is documented and reported to the client. The Case Narrative Report is filed with the data and is also useful for production of case narratives that are issued with the final data reports. If a problem exists that requires follow-up to rectify, a Corrective Action Report (CAR) is issued to document the problem found, steps taken to resolve the problem, and what samples were affected. This CAR form is filed by the Quality Manager and reviewed by management to verify that appropriate actions have been taken to correct the problem.

## 16. Corrective Actions for Out-of-Control Data

16.1. The Table below outlines the data assessment, acceptance criteria, and corrective action procedures for out-of-control data:

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration	The five point calibration is analyzed initially and when Continuing Calibration Check standard fails criteria.	- %RSD $\leq$ 20% for the relative response factors for the calibration standards if using average response factor calibration. Correlation Coefficient R must be $>0.990$ for Linear Regression.	- Re-analyze the initial calibration standard and/or evaluate/correct instrument malfunction to obtain initial calibration and continuing calibration check standards that meet criteria.
Continuing Calibration Check Standard (CCCS)	<ul style="list-style-type: none"> <li>- Initially analyze a CCCS immediately following an initial calibration.</li> <li>- After the initial CCCS of the sequence, a CCCS must be analyzed after 9 samples.</li> <li>- Analytical sequence must end with analysis of a CCCS.</li> </ul>	<ul style="list-style-type: none"> <li>- Calibration factor for the continuing calibration check must <math>\pm 15\%</math> of the true value.</li> <li>- Retention time of all quantitated peaks must be within RT window (reset with each initial CCCS of a sequence).</li> <li>- All samples must be bracketed by a CCCS that meet all criteria stated above.</li> </ul>	<ul style="list-style-type: none"> <li>- If the reason for the failure of the CCCS appears to be a poor injection (or a degraded standard solution), the CCCS will be re-injected (or re-prepared and re-injected) immediately following the failed CCCS. This can only occur if the instrument is being attended by an analyst. If upon re-injection, the CCCS meets all the acceptance criteria and there is no apparent impact on the sample data the analytical sequence will continue and samples will not be reanalyzed. The associated sample data will be reported.</li> <li>- If CCCS failure was not due to a poor injection (or degraded</li> </ul>

			<p>standard solution) or the instrument was unattended at the time of the CCCS failure, correct system, if necessary, and recalibrate. Initial calibration and CCS criteria must be met before sample analysis may begin. Samples that are not bracketed by complaint CCCSs must be reanalyzed.</p> <p>-If acceptable CCCSs are observed later in the sequence, samples bracketed by acceptable CCCSs will be reported. Samples between the failed CCS and prior/ subsequent complaint CCCS will be re-analyzed.</p>
-Retention Time (RT)	<p>- Use the retention time for peak in the CCSs to determine midpoint of the relative retention time window for the analysis sequence.</p> <p>-Each sample analysis: Rely on RT windows to identify PCB Aroclor to report. Also use pattern recognition and professional judgment for peaks that shift from RT windows, because compound composition may shift RT for GC peaks.</p>	- Each quantitated peak and surrogate peak should be within established windows.	-Inspect chromatographic system for malfunction, correct problem. Perform re-analysis if necessary.
Method Blank	<p>-One per extraction batch of ≤20 samples of the same matrix per day.</p> <p>-Must be analyzed on each instrument used to analyze associated samples.</p> <p>-Must undergo all sample preparative procedures.</p>	<p>- Concentration does not exceed the RL for any PCB Aroclor.</p> <p>- Surrogates must meet in required limits of 60-120%.</p>	<p>- Re-analyze method blank to determine if instrument contamination was the cause. If method blank re-analysis passes, then report samples.</p> <p>-If method blank is found to contain PCB contamination above the RL for any PCB Aroclor compound, then re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data B flagged to indicate method blank contamination.</p>

Laboratory Control Sample (LCS)	- One per extraction batch of ≤20 samples per matrix per day.	-Percent recovery must be within method limits. - Must meet Aroclor spike criteria of 70-130% recovery - Surrogates must meet require limits of 60-120%.	-Re-analyze LCS to determine if instrument was the cause. If LCS passes, then report samples. -If LCS recovery is still out of limits, the re-extract and re-analyze all associated samples. If no sample exists for re-extraction, report data flagged to indicate LCS failed recovery.
Surrogates	-Calibrated as target compounds in the Aroclor A1254 Standards. -Surrogates are added to all calibration check standards, blanks (including instrument blanks run after CCCS), samples and QC samples.	- Surrogates must meet require limits of 60-120%.	-Re-analyze the affected sample or QC sample to determine if instrument was the cause. If surrogate passes, then report samples. -Check for errors in surrogate calculation and surrogate solutions. -If no problem is found, then re-extract and re-analyze the sample. -If re-extraction is within limits and sample extract holding time, then report only the re-analysis. -If the re-extraction is within limits, but out of extraction holding time, then report both sets of data. -If the re-extraction produces surrogate recovery still out of limits, then report both sets of data. -If no sample exists for re-extraction, report data flagged to indicate surrogate failed recovery or have a client re-sample.

**17. Contingencies for Handling Out-of-Control or Unacceptable Data**

17.1. Data that is detected to be out-of-control for any reason, when compared to method acceptance criteria, will be addressed in the following manner:

17.1.1. If the problem exists with the gas chromatographic instrumentation, appropriate action will be taken to repair and perform maintenance to bring the instrument back to operating condition. Once the instrumentation is determined to be correctly operating analysis can begin again.

17.1.2. If the problem exists with calibration standard solutions, the analyst will prepare new standards and discard the standard solutions that are suspect. Instrument calibration can be performed, and analysis can begin, once system is in control.

17.1.3. If the problem exists with sample extraction and extract preparation, the extraction step that is producing the out-of-control situation will be diagnosed and rectified. Once the troubleshooting procedures correct the problem, extraction can once again occur, and analysis can continue.

17.2. In situations where data is reported under out-of-control conditions, the data will be annotated with data qualifiers and/or appropriate descriptive comments defining the nature of the excursion in the sample case narrative. If warranted, a corrective action report (CAR) will be issued to define the problem, steps to correct the problem, and final resolution. The Quality Manager and client must be notified.

## 18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. Initial Demonstration of Capability (IDOC) Procedure:

18.2.1. Prepare 4 replicates of a fortified laboratory blank sample by spiking each PUF sample with 1.0mL of 1.00ug/mL Aroclor solution. The Aroclor type used for spiking should be rotated on a yearly basis. Prepare one method blank sample with the batch.

18.2.2. For each replicate the recovery value of the sample must fall in the range of 70-130% (or established lab limits) and the percent RSD must be <20 % for the method performance to be considered acceptable.

18.2.3. This procedure must be repeated using four fresh samples until satisfactory performance has been demonstrated. The initial demonstration of capability is used primarily to preclude the laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it. It is expected that as laboratory personnel gain experience with this method the quality of data will improve beyond those required here.

18.3. Continuing Demonstration of Performance Procedure:

18.3.1. Annual continuing demonstration of performance may be satisfied by a repeat Initial Demonstration of Performance, the acceptable analysis of an unknown samples (for example PT test sample), or the acceptable analysis of 4 consecutive Laboratory Control Spike samples. Records of continuing demonstration of performance are maintained by the laboratory Quality Assurance Department.

18.3.2. With each batch of samples to be extracted a method blank is processed. The method blank is carried through all stages of sample preparation and measurement steps. For water samples an organic-free reagent water blank is processed.

18.3.3. The method blank should exhibit PCB levels less than the practical quantification limit or reporting limit (PQL or RL). If the method blank exhibits PCB contamination above the reportable quantitation limit, the samples associated with the contaminated blank should be re-extracted and analysis repeated when appropriate. If there is no original sample available for re-extraction or if the associated sample concentrations greatly exceed the blank concentration, then all positive concentration results for the associated samples should be flagged with a "B" indicating blank contamination and a case narrative describing the situation prepared.

18.3.4. A matrix spike/ matrix spike duplicate is to be analyzed at a rate of 1 matrix spike/ matrix spike duplicate per every 20 samples. A duplicate sample may be prepared in lieu of a matrix spike duplicate in place of a matrix spike duplicate when detectable PCB concentrations are known to be present.

#### 18.4. Method Detection Limit:

18.4.1. A method detection limit will be determined for this method whenever major modification to the extraction or analysis procedures are made or at a minimum frequency of every 2 years. A minimum of seven laboratory organic free water samples or sodium sulfate will be prepared and spiked with chlorinated PCB methyl esters mixture, at a low level and taken through all extraction and analytical procedures.

$$MDL = S * t_{(n-1, 1-\alpha=0.99)}$$

S = Standard deviation of the replicate analyses

n = Number of replicates

$t_{(n-1, 1-\alpha=0.99)}$  = Student's t value for the 99% confidence level with n-1

For example: t for 8 replicates =  $t_{(7,0.99)} = 2.998$

18.4.2. The determined MDL must be less than the concentration spiked but greater than one tenth (1/10) the spiked concentration. If not, repeat the MDL determination at an appropriate spike concentration for affected analytes.

### 19. Method Modifications

19.1. Not applicable to this SOP.

### 20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

### 21. Troubleshooting

21.1. Not applicable to this SOP.

### 22. Safety

22.1. Safety glasses, a lab coat, and disposable gloves must be worn when handling samples and extracts.

22.2. All manipulations of samples should be conducted inside a chemical fume hood. Manipulation of sample extracts outside of a fume hood should be minimized by the analyst.

22.3. Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of safety data sheets to familiarize themselves with the precautions for handling solvents and chemicals used to process samples. The analyst should refer to the laboratory chemical hygiene plan for further safety information.

22.4. Samples remaining after analysis should either be returned to the customer for disposal or disposed of through the laboratory's disposal plan. Refer to the sample custodian for assistance and also SOP S-NY-S-054, disposal of laboratory waste.

### **23. Waste Management**

23.1. All applicable federal and state rules and regulations governing hazardous waste will be followed when disposing of laboratory waste generated during the execution of this method.

23.2. Please refer to standard operating procedure, S-NY-S-054, regarding how hazardous waste is handled and disposed of by the laboratory.

### **24. Pollution Prevention**

24.1. Pollution prevention is practiced in the laboratory by minimizing usage of solvents and chemicals, so that disposal of waste generated is held to the smallest amount possible. This is directly linked to the types of extraction procedures in place at the laboratory to reduce the volumes of solvents.

24.2. Pollution prevention also relies on minimizing to the best extent the chemicals and solvents required to perform extraction and analysis procedures. The laboratory personnel strive to purchase chemicals and standards that will be consumed based on anticipated workload. For additional information about laboratory pollution prevention, please refer to laboratory SOP S-NY-S-168.

### **25. References**

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. U.S. EPA Method TO-4A "Determination of Pesticide and Polychlorinated Biphenyls in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gas Chromatographic/Multi-Detector Detection (GC/MD).

25.5. U.S. EPA Method TO-10A "Determination of Pesticides and Polychlorinated Biphenyls in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector (GC/MD) Detection.

25.6. U.S. EPA SW-846 Method 8082A "Test Methods for Evaluating Solid waste; Volume 1B Laboratory Manual Physical/Chemical Methods", Office of Solid Waste and Emergency Response, Third Edition, Final Update III, December 1996.

25.7. U.S. EPA 40 CFP Part 136, "Guidelines Establishing Test Procedures of the Analysis of Pollutants", July, 1988.

25.8. Standard Methods for the Examination of Water and Waste Water", 19<sup>th</sup> Edition 1995, American Public Health Association, American Water Works Association, Water Pollution Control Federation.

25.9. New York State Department of Health, "Environmental Laboratory Approval Program Certification Manual", Wadsworth Center for laboratories and Research, 1996.

25.10. Guide to Environmental Analytical Methods", third edition, Genium Publishing Corporation, 1997.

## 26. Tables, Diagrams, Flowcharts, and Validation Data

- 26.1. Attachment I: PCB Stock Standard/Calibration Standard Preparation.
- 26.2. Attachment II: Continuing Calibration Check Standard Preparation.
- 26.3. Attachment III: GC Operating Parameters.

## 27. Revisions

Document Number	Reason for Change	Date
S-NY-O-341-rev.00	First Issue.	03March2015
S-NY-O-341-rev.01	Section 9.1: Updated instrument used Sections 13.4 and 16.1: Updated surrogate recovery limits	24March2015
S-NY-O-341-rev.02	Sections 13.8.2.1 and 14.1: added documentation regarding quantitation using 3-4 peaks	30March2015

## Attachment I: PCB Stock Standard/Calibration Standard Preparation

**Table 1: PCB Stock Standard Preparation Table**

PCB Stock Standards	1. Init. Volume (mL)	Final Volume (mL)	Conc. (ppm)
A1016	5.0	50	10.0
A1221	5.0	50	10.0
A1232	5.0	50	10.0
A1242	5.0	50	10.0
A1248	5.0	50	10.0
A1254	5.0	50	10.0
A1260	5.0	50	10.0
A1262	1.0	100	10.0
A1268	1.0	100	10.0

Unless otherwise noted hexane is the solution used to make all dilutions. \*Custom Order

**Table 2: PCB Calibration Standard Preparation Table (High Level Calibration Curve)**

Initial Volume (mL)	Initial Conc. (ug/mL)	Final Volume (mL)	Final Concentration (ppm)					
			A1016	A1221	A1232	A1242	A1248	A1260
5.0	(10.0)	50.0	1.000	1.000	1.000	1.000	1.000	1.000
2.5	(10.0)	50.0	0.500	0.500	0.500	0.500	0.500	0.500
1.25	(10.0)	50.0	0.250	0.250	0.250	0.250	0.250	0.250
1.00	(10.0)	50.0	0.200	0.200	0.200	0.200	0.200	0.200
0.500	(10.0)	50.0	0.100	0.100	0.100	0.100	0.100	0.100
5.0	(1.00)	50.0	0.020	0.020	0.020	0.020	0.020	0.020

Actual Concentration, see Table 1 for actual working standard concentrations for each Aroclor.  
 See Table 3 for A1254 Standard Preparation (high level).

**Attachment I: PCB Stock Standard/Calibration Standard Preparation (continued)**

**Table 2A: PCB Calibration Standard Preparation Table (Low Level Calibration Curve)**

Init. Volume (mL)	Initial Conc. (ug/ml)	Final Volume (mL)	Final Concentration (PPM)					
			A1016	A1221	A1232	A1242	A1248	A1260
0.5	(10.0)	50.0	0.100	0.100	0.100	0.100	0.100	0.100
2.5	(1.0)	50.0	0.050	0.050	0.050	0.050	0.050	0.050
1.0	(1.0)	50.0	0.020	0.020	0.020	0.020	0.020	0.020
1.0	(0.500)	50.0	0.010	0.010	0.010	0.010	0.010	0.010
0.50	(0.500)	50.0	0.005	0.005	0.005	0.005	0.005	0.005

Actual Concentration, see Tables 1 and 2 for actual working standard concentrations for each Aroclor.  
 See Table 3A for A1254 Standard Preparation (low level).

**Table 3: PCB A1254 Calibration Standard Preparation Table (for High Level Curve)**

Initial Volume (mL) A1254	Initial Conc. (ug/mL) A1254	Initial Volume (mL) 0.5/5.0 -PPM Surrogate	Final Volume (mL)	Final Concentration (PPM)		
				A1254	TCMX	DCBP
5.0	10.0	0	50	1.000	0	0
2.5	10.0	0	50	0.500	0	0
5.0	10.00	2.00	50	1.000	0.020	0.200
2.5	10.0	1.00	50	0.500	0.010	0.100
1.25	10.0	0.800	50	0.250	0.008	0.080
0.500	10.0	0.500	50	0.100	0.005	0.050
1.000**	1.000	0.200	50	0.020	0.002	0.020

\*\*This initial volume is of the A1254 1.000ppm secondary stock solution WITHOUT surrogates.

**Attachment I: PCB Stock Standard/Calibration Standard Preparation (continued)**

**Table 3A: PCB A1254, TCMX and DCBP Calibration Standard Preparation Table (for Low Level Curve)**

Initial Volume A1254 (mL)	Initial Conc. A1254 (ug/mL)	Initial Volume (mL) 0.5/5.0 -PPM Surrogate	Final Volume (mL)	Final Concentration (PPM)		
				A1254	TCMX	DCBP
0.5	10.0	0.80	50	0.100	0.00800	0.0800
2.50	1.000	0.50	50	0.050	0.00500	0.0500
1.0	1.000	0.40	50	0.020	0.00400	0.0400
1.0	0.500	0.250	50	0.010	0.00250	0.0250
0.50	0.500	0.100	50	0.005	0.00100	0.0100

## Attachment II: Continuing Calibration Check Standard Preparation

**Table 1: PCB Continuing Calibration Working Standards prepared from 1000ppm Stock Standards**

<b>PCB</b>	<b>Initial Volume (mL)</b>	<b>Final Volume (mL)</b>	<b>Concentration (ppm)</b>
A1016	1.0	100	10.0
A1221	1.0	100	10.0
A1232	1.0	100	10.0
A1242	1.0	100	10.0
A1248	1.0	100	10.0
A1254	1.0	100	10.0
A1260	1.0	100	10.0

## Attachment II: Continuing Calibration Check Standard Preparation (continued)

**Table 2: PCB Continuing Calibration Standards (High Level) prepared from 10ppm CCV Working Standards and all contain surrogates**

PCB	Surr. Volume* (mL)	Initial Volume (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (ppm)	Aroclor Concentration (ppm)
A1016	2.0	5.0	100	0.010/0.100	0.500
A1221	2.0	5.0	100	0.010/0.100	0.500
A1232	2.0	5.0	100	0.010/0.100	0.500
A1242	2.0	5.0	100	0.010/0.100	0.500
A1248	2.0	5.0	100	0.010/0.100	0.500
A1254	2.0	5.0	100	0.010/0.100	0.500
A1260	2.0	5.0	100	0.010/0.100	0.500

\*Surrogate stock solution 0.500ppm TCMX and 5.0ppm DCBP.

**Table 3: PCB Continuing Calibration Standards (low Level) prepared from 10.0ppm CCV Working Standards and all contain surrogates**

PCB	Surr. Volume* (mL)	Initial Volume (mL)	Final Volume (mL)	Surrogate Concentration TCMX/DCBP (PPM)	Aroclor Concentration (PPM)
A1016	1.0	0.500	100	0.005/0.050	0.050
A1221	1.0	0.500	100	0.005/0.050	0.050
A1232	1.0	0.500	100	0.005/0.050	0.050
A1242	1.0	0.500	100	0.005/0.050	0.050
A1248	1.0	0.500	100	0.005/0.050	0.050
A1254	1.0	0.500	100	0.005/0.050	0.050
A1260	1.0	0.500	100	0.005/0.050	0.050

\*Surrogate stock solution 0.500ppm TCMX and 5.0ppm DCBP.

## Attachment III: GC Operating Parameters

GC #: GC-21 8082 High Level Method (GEHR inclusive, parameters)  
 Method: Method 2  
 Column: ZB-1 Front  
 ZB-5 Middle  
 Date: 01/18/2012  
 Analyst: JKA  
 File Name: S:\Lab Data\PCB\GC Parameters [GC21\_Parameters.xls]8082.M2

**Sample Delivery:** SEE LEAP PARAMETERS

**Column Oven:**

Step	Temp (°C)	Rate (°C/min)	Hold (min)	Total (min)
Initial	140	-----	2.00	2.00
2	200	10	0.00	8.00
3	245	5	13.5	30.5

Stabilization Time (min): 0.20

**Injector: Front CP-1177**

1177 Oven Power: ON  
 1177 Temperature (°C): 300

Time	Split State	Split Ratio
Initial	ON	30

**Flow/PSI(Front EFC, Type 1):**

Carrier Gas: Helium

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30.0*	-----	20	20

Constant Flow Mode Enable: NO  
 Column Flow Rate (ml/min): 5.0

**Detector: Front ECD**

ECD Oven Power: ON  
 Temperature (°C): 300  
 Electronics: ON  
 Range: 1

Time	Range	Autorezo
Initial	1	YES

Front ECD Adjustment  
 Time Constant: Fast  
 Cell Current: CAP  
 Contact Potential (mV): -650 \*  
 Date of last adjustment: 12/15/2011

**Front ECD Adjustments:**  
 Make-up Flow (mL/min): 35 \*  
 \*values may change with use

**Analog Output**

Detectors: Front: ECD Attenuation 1  
 Middle: ECD Attenuation 1  
 Rear: None

**Injector: Middle CP-1177**

1177 Oven Power: ON  
 1177 Temperature (°C): 300

Time	Split State	Split Ratio
Initial	ON	30

**Flow/PSI(Front EFC, Type 1):**

Step	Pres (psi)	Rate (psi/min)	Hold (min)	Total (min)
Initial	30.0*	-----	20	20

Constant Flow Mode Enable: NO  
 Column Flow Rate (ml/min): 5.0

**Middle ECD**

ECD Oven Power: ON  
 Temperature (°C): 300  
 Electronics: ON  
 Range: 1

Time	Range	Autorezo
Initial	1	YES

Middle ECD Adjustment  
 Time Constant: Fast  
 Cell Current: CAP  
 Contact Potential (mV): -380 \*  
 Date of last adjustment: 12/15/2011

**Middle ECD Adjustments:**  
 Make-up Flow (mL/min): 35 \*

## **Appendix C**

Anchor Bond Anchor  
Crete System  
Specifications



## ANCHOR BOND ANCHOR CRETE SYSTEM



Anchor Crete is a urethane fortified cementitious coating, trowel applied at 1/4" to 1/8". This system provides protection against harsh chemical attack as well as the stresses caused by thermal shock and high impact

We offer this flooring system in three standard colors: gray, red and black.

Anchor Crete's finished texture provides a slip-resistant yet cleanable surface.

PROPERTIES	TEST METHOD	ANCHOR CRETE 1/4"	ANCHOR CRETE SL
COMPRESSIVE STRENGTH	ASTM C 579	7,300 psi	6,700 psi
		50.3 MPa	46.2 MPa
FLEXURAL STRENGTH	ASTM C 580	1,800 psi	2,600 psi
		12.4 MPa	17.9 MPa
TENSILE STRENGTH	ASTM C 307	800 psi	1,000 psi
		5.5 MPa	6.9 MPa
MODULUS OF ELASTICITY	ASTM C 580	1.7x 10 <sup>-5</sup> psi	1.5x 10 <sup>-5</sup> psi
		1170 MPa	1030 MPa
COEFFICIENT OF THERMAL EXPANSION	ASTM C 531	1.1x 10 <sup>-5</sup> °F	2.2x 10 <sup>-5</sup> °F
		2.0x 10 <sup>-5</sup> °C	4.0x 10 <sup>-5</sup> °C
WATER ABSORPTION	ASTM C 413	<0.1%	<0.1%
THERMAL CONDUCTIVITY	ASTM C 179	8 Btu in./Ft <sup>2</sup> -h-°F	6 Btu in./Ft <sup>2</sup> -h-°F
		1.2 W/mK	.9 W/mK
DENSITY	ASTM C 905	130 lb/ft <sup>3</sup>	123 lb/ft <sup>3</sup>
		2.08 g/cm <sup>3</sup>	1.97 g/cm <sup>3</sup>
RESISTANCE TO FUNGI GROWTH	* ASTM G 21	1	1

\* Scale of 1 to 4, 1 Being least growth

**STANDS UP TO AGGRESSIVE CLEANING TECHNIQUES:** ANCHOR CRETE Systems can be exposed to severe daily cleaning techniques, including live steam, hot water, aggressive detergents and disinfectants

**OFFERS PROTECTION FROM THERMAL SHOCK:** ANCHOR CRETE is unaffected by freeze/thaw cycles and withstands extreme temperature ranges while in service and during cleaning. Unlike other flooring systems, ANCHOR CRETE won't delaminate due to thermally induced stresses. Will withstand temperatures ranging from -50°F to +250°F.

**CHEMICAL RESISTANT:** ANCHOR CRETE Systems are resistant to damage from caustics, organic and inorganic acids, solvents and most other commonly used chemicals

**ADVANTAGES**  
HIGHLY IMPACT / ABRASION RESISTANT  
CHEMICAL RESISTANT  
THERMAL SHOCK RESISTANT

**APPLICATIONS**  
CIP ROOMS  
PRODUCTION ROOMS  
COOLERS / FREEZERS  
COOL ENVIRONMENTS WITH HOT WATER CLEAN UP